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# IMIDE MODIFIED EPOXY MATRIX RESINS

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Final Report
Contract NAS3-22032

Prepared for
NASA Lewis Research Center
Cleveland, Ohio 44135

by


D.A. Scola
R.H. Pater


February 1, 1981

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16. Abstract This report describes the results of a ten-month program designed to develop high char yield epoxy resins using novel bisimide amines (BIA's) as curing agents with a state-of-the-art epoxy resin, MY 720. Four bisimide amines were evaluated in this program. These were the BIA's derived from the 6F anhydride (4,4'-hexafluoroisopropylidene biphthalic anhydride) and the aromatic diamines, 4,4'-diaminodiphenylsulfone (DDS), 4,4'-methylenedianiline (MDA), 4,4'-oxydianiline (ODA) and 4,4'-paraphenylenediamine (PDA). The BIA's were abbreviated 6F-DDS, 6F-MDA, 6F-ODA and 6F-PDA, corresponding to the 6F anhydride and diamines mentioned above. Stoichiometric quantities of the epoxy resin and the BIA's (MY 720/6F-DDS, MY 720/6F-MDA, MY 720/6F-ODA and MY 720/6F-PDA) and a 50:50 mixture of a BIA and the parent diamine (MY 720/6F-DDS/DDS, MY 720/6F-MDA/MDA, MY 720/6F-ODA/ODA and MY 720/6F-PDA/PDA) were studied to determine the cure cycle required for preparation of resin specimens. The bisimide cured epoxies were designated IME's (for imide modified epoxy). The physical, thermal and mechanical properties of these novel resins were determined. The levels of moisture absorption exhibited by the bisimide amine cured epoxies (IME's) were considerably lower than the state-of-the-art epoxies. The strain-to-failure of the control resin system (MY 720/DDS) was improved 25% by replacement of DDS with 6F-DDS (MY 720/6F-DDS). Each BIA-containing resin exhibited twice the char yield of the control resin MY 720/DDS (40% char vs 20% char). Graphite fiber reinforced control (C) and IME resins were fabricated and characterized. Two of the composite systems, Celion 6000/IME-1 and Celion 6000/IME-5 (where IME-1 is MY 720 cured with the BIA 6F-DDS and IME-5 is MY 720 cured with a 50:50 mixture of DDS and the BIA 6F-DDS) emerged as having superior properties compared to the other Celion 6000/IME composite systems and also compared to state-of-the-art graphite epoxy systems. These two systems exhibited excellent wet shear and flexural strengths and moduli at 300 and 350°F.

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Imide Modified Epoxy Matrix Resin

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## Imide Modified Epoxy Matrix Resin

### SUMMARY

This report describes the results of a ten-month program designed to develop tough, high char yield epoxy resins using novel bisimide amines (BIA's) as curing agents with a state-of-the-art epoxy resin, MY 720. Four bisimide amines were evaluated in this program. These were the BIA's derived from the 6F anhydride (4,4'-hexafluoroisopropylidene biphthalic anhydride) and the aromatic diamines, 4,4'-diaminodiphenylsulfone (DDS), 4,4'-methylenedianiline (MDA), 4,4'-oxydianiline (ODA) and 4,4'-paraphenylenediamine (PDA). The BIA's were abbreviated 6F-DDS, 6F-MDA, 6F-ODA and 6F-PDA, corresponding to the 6F anhydride and diamines mentioned above. Stoichiometric quantities of the epoxy resin and the BIA's (MY 720/6F-DDS, MY 720/6F-MDA, MY 720/6F-ODA and MY 720/6F-PDA) and a 50:50 mixture of a BIA and the parent diamine (MY 720/6F-DDS/DDS, MY 720/6F-MDA/MDA, MY 720/6F-ODA/ODA and MY 720/6F-PDA/PDA) were studied to determine the cure cycle required for preparation of resin specimens. The bisimide cured epoxies were designed IME's (for imide modified epoxy). The physical, thermal and mechanical properties of these novel resins were determined. The levels of moisture absorption exhibited by these bisimide amine cured epoxies (IME's) were considerably lower than the state-of-the-art epoxies. The strain-to-failure of the control resin system (MY 720/DDS) was improved 25% by replacement of DDS with 6F-DDS (MY 720/6F-DDS). All of the IME resins exhibited twice the char yield of the control resin (40% char vs 20% char). Graphite fiber reinforced control (C) and IME resins were fabricated and characterized. Two of the composite systems, Celion 6000/IME-1 and Celion 6000/IME-5 where IME-1 is MY 720 cured with the BIA 6F-DDS and IME-5 is MY 720 cured with a 50:50 mixture of DDS and the BIA 6F-DDS emerged as having superior properties compared to the other Celion 6000/IME composite systems and also compared to state-of-the-art graphite epoxy systems. These two systems exhibited excellent wet shear and flexural strengths and moduli at 300 and 350°F.

# 1. INTRODUCTION

This document constitutes the final report on a ten (10) month program to characterize novel imide modified epoxy (IME) resins cured by bisimide amine hardeners, newly developed by investigators at NASA Lewis (Refs. 1,2). State-of-the-art epoxy resins are finding wide applications as matrices in advanced fiber reinforced resin composites which are being used in aerospace vehicles as primary and secondary structural components. However, the brittle nature and poor char forming characteristics of state-of-the-art epoxy resins dictate that changes in molecular structure are necessary to improve toughness and increase char forming ability. Toughness is required for resin matrices used in aerospace structures where high strain-to-failure, durability, and reliability are of prime importance. Higher char yield epoxy resins are desirable because they would provide composites which exhibit improved retention of structural integrity in the event the composite is subjected to a fire.

## 1.1 Objective of the Program

The objective of this program was to characterize novel imide modified epoxy resins cured by novel bisimide amine hardeners.

## 1.2 Program Tasks

The work performed under this contract was accomplished in three (3) technical tasks as follows:

Task I. Comprised preparation and characterization of nine epoxy resin systems which were prepared from tetraglycidyl methylenedianiline (MY 720) cured with a stoichiometric quantity of bisimide-amine and aromatic diamine hardeners. The physical and mechanical properties as well as char forming characteristics were determined.

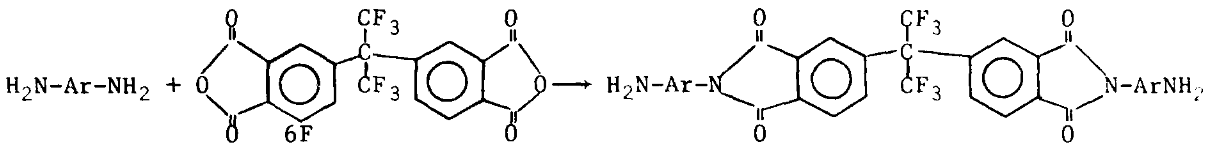
Task II. In this task, the prepreg and laminate processing parameters were investigated.

Task III. The mechanical properties of the novel epoxy resins reinforced with Celion 6000 graphite fibers were determined in the dry condition and after exposure to 87% RH at 82°C to saturation. The physical properties such as glass transition temperature, density, fiber, resin and void content and fiber distribution of the laminates were also determined. In addition, nine (9) unidirectional laminates were delivered to the NASA Project Manager.

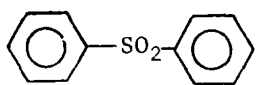
## 2. RESULTS AND DISCUSSION

### 2.1 Synthesis of Bisimide Amines

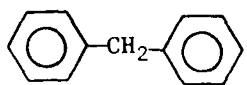
The structures of the bisimide amines (BIA's), the aromatic diamines from which the BIA's were synthesized and the base epoxy resin used in this program are listed in Table 1. The synthetic route used in preparing the BIA's is represented by this general equation (1)



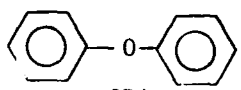

(1)

where Ar =  , the BIA is designated 6F-DDS

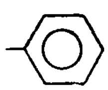
DDS

=  , the BIA is designated 6F-MDA

MDA

=  , the BIA is designated 6F-ODA

ODA

=  , the BIA is designated 6F-PDA

PDA

The general procedure used in the BIA preparations consisted of refluxing a solution of the aromatic diamine (0.48 mole) and 6F (0.24 mole) in NMP for 4 hrs. The reaction mixture was then concentrated to half its volume, and poured into an ice-water mixture to yield a crude 6F-diamine product, which was washed several times with hot distilled water. The crude products were recrystallized from hot acetone-water mixture in high yields (90% or better). Elemental analyses, infrared and nuclear magnetic resonance (NMR) spectra were consistent for each product. Comparison of the differential scanning calorimetry (DSC) thermograms, NMR and infrared (IR) spectra with the DSC, NMR and IR's of BIA's purchased from DuPont showed that these materials were almost identical. The GPC chromatograms of the UTRC prepared BIA's and of the DuPont BIA's are compared in Figs. 1 and 2. The IR's, NMR's and DSC's of each BIA synthesized in this study are shown in Figs. 3 through 5.

## 2.2 Composition of Bisimide Amine Cured MY 720 Epoxy Resins

Table 1 shows the structures of the uncured epoxy resin and curing agents used in preparing the epoxy resin specimens and fiber reinforced graphite imide modified cured epoxy resin composites. The designation for each material is also listed in Table 1. The composition of each resin system, including the molecular weight of each component, the mole ratio of resin to hardener(s) used throughout the study and the epoxy resin system designation are listed in Table 2. The actual weights of epoxy to hardener(s) used in the initial studies to determine the cure cycle of each system and to fabricate small resin specimens are also listed in Table 2.

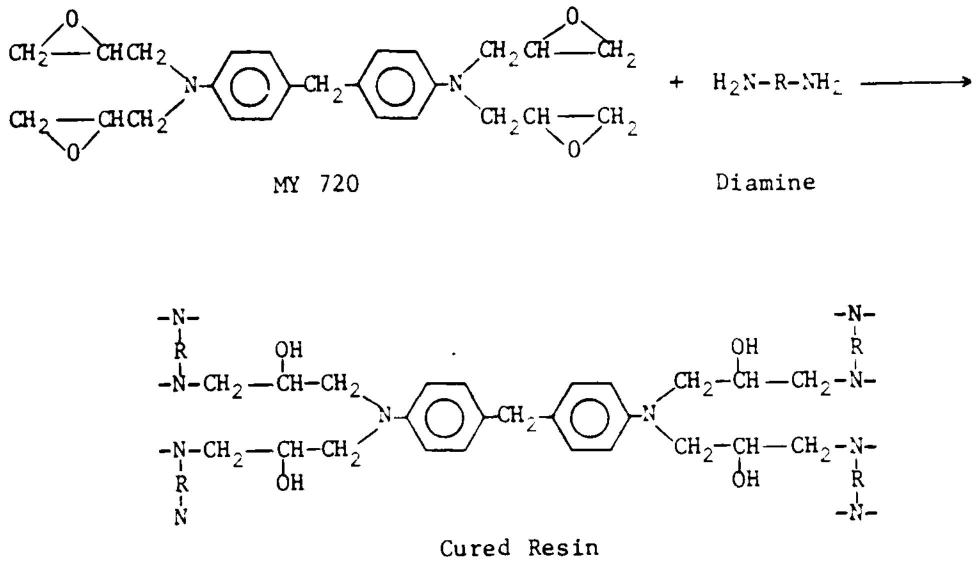
## 2.3 Determination of Resin Thermal Behavior and Cure Cycle

The gel times and time/temperature cure relationship for each resin system (listed in Table 2) were determined. Homogeneous mixtures of the resin systems were required to determine the cure cycle. Two approaches were tested to obtain a homogeneous mixture of the curing agent and MY 720 epoxy resin. One involved the use of acetone as solvent to dissolve the mixture followed by removal of the solvent to give a solventless homogeneous powder mixture. The other approach involved heating the mixture at 120°C for 15 min to give a liquefied mixture. The solvent approach worked well for all of the nine resin systems. The second method, however, was only applicable to the MY 720/DDS resin system. Its application to the other eight resin systems involving the use of the bisimide-amine hardeners failed because of the high melting points and insolubility of the bisimide amines. Thus, the solvent method was used to mix the curing agent and epoxy resin in this study.

### 2.3.1 Infrared (IR) Study of Cure Reaction

The cure reaction of the nine resin systems whose compositions are given in Table 3 was studied by infrared spectroscopy. Each of the solventless and homogeneous epoxy-amine mixtures was cured in three stages at 122°C, 177°C, and 202°C between two sodium chloride salt plates for intervals of 1 hr, or longer, and the IR spectra after each time interval were recorded.

The cure process and rates of cure of the resin systems were followed by measuring the disappearance of the absorbance at 903 $cm^{-1}$, assigned to a fundamental vibration of the epoxy ring of MY 720. Alternatively, the cure process and rates of the cure reaction can also be measured by following the appearance of the absorbance at 3390 $cm^{-1}$, attributable to the stretching vibration of an OH group formed from the reaction of the epoxy ring of MY 720 with the amine group of the curing agents. The basic cure reaction for these resins is given in Eq. (2).




(2)

Because there is some evidence to indicate that the hydroxyl group also reacts with the epoxy group to form ether groups, the change in absorption due to hydroxyl is not a good measure of the cure rate or degree of cure of the resin. Therefore only the absorption due to the epoxy group at 903 $cm^{-1}$ was followed in this study. Assuming that the MY 720 phenyl group absorption band at 1512 $cm^{-1}$ is constant, all 903 $cm^{-1}$ absorbances were expressed as a ratio of the absorbance at 903 $cm^{-1}$ to the absorbance at 1512 $cm^{-1}$ and were plotted as a function of cure time to establish the degree and rate of cure for each resin system. The absorption ratios are listed in Table 4. Plots of the absorption ratios as a function of time at given temperatures are given in Figs. 6a and b for all the resin systems. An example of the changes in the IR which occur as a function of temperature and time for the entire cure study is given in Fig. 7 for the IME-1 resin system. Infrared spectra for the other IME resin systems at RT and after virtually complete cure are given in Figs. 8-15.

Inspection of Fig. 7 reveals that a rapid chemical conversion occurred at 122°C for 1 hr followed by 177°C for another hour. Little change occurred upon further curing at 177°C from 2 to 5 hrs, but after treatment at 202°C for up to 2.5 hrs, almost complete disappearance of the epoxy group appears to have occurred. Postcuring for 24 hrs does not appear to reduce the absorption due to the epoxy group relative to the 2.5 hr postcure. All of the resin systems required similar treatment for complete cure, but systems IME-6, 7 and 8 exhibited greater room temperature reactivity than the other IME resin systems. This is illustrated by the low values of the absorption ratio at room temperature for these resins. The graphs of absorption ratios (epoxy/aromatic) as a function of time at given temperatures (Figs. 6a & 6b) reveal some unique differences in these IME resin systems. Resin systems C and IME-1 which have the sulfone amine functionality in the curing agent are less reactive than the other IME systems which are derived from MDA, ODA and PDA. Another significant point, is that these latter systems appear to have similar reactivity with IME-3 which is perhaps the most reactive of this series. This is also true for IME-7 which contains 6F-ODA and ODA.

Figures 6a and 6b further show that for most of the IME resins no differences in the cure state exists after 2 hrs or 5 hrs at 177°C, suggesting that only 2 hrs is required at this temperature and further suggesting that higher temperatures are needed for additional cure. A continuous slow cure appears to occur in the IME-2 and -3, -7 and -8 resin systems at this temperature. Figures 6a & 6b also show that after 2.5 hrs at 202°C, cure is virtually complete, since the absorption ratio of epoxy/aromatic is approaching zero and very little change in this ratio occurs after 24 hrs at 202°C. Although the IR studies suggest that treatment at 202°C for 2.5 hrs causes complete cure, to obtain optimum 150°C and 177°C shear and flexural properties (section 2.5.7) a 24 hr postcure was required.

### 2.3.2 Differential Scanning Calorimetry (DSC) Studies

The DSC thermal behavior of the nine uncured MY 720/amine mixtures are shown in Figs. 16a-i, and the data are summarized in Table 5.

Generally the uncured resins exhibit two major exotherms in the temperature range between 25° to 300°C, which are most likely related to the cure process. A third exotherm occurs above 300°C. This is related to decomposition of the cured resins. This view is supported by the thermogravimetric (TGA) studies described in Section 2.4.3. In each case, a weight loss is initiated at about 290°C and becomes significant between 300° to 450°C. Based on the DSC behavior IME-1 and -5 resin systems appear to exhibit similar cure characteristics. The first peak exotherm in both cases is only 120°C compared to approximately 200°C for IME-2, -3, -4, -6 and -7, and the second peak exotherm for IME-1 and -5 is also lower than the peak exotherm of the above mentioned resins. Resins C and IME-8 exhibit similar DSC behavior. In resins C and IME-8 the first exotherm peak appears at 155°C, lower than the other resin systems, except for IME-1 and -5. Moreover, the second exotherm peak for these two systems is also lower than the other resins, (except for IME-1 and -5). The DSC behavior of the IME-8 system is not expected in view of the DSC characteristics of IME-8 which contains 6F-PDA, while IME-8 contains 6F-PDA and PDA. This suggests that 6F-PDA is more reactive than PDA. An alternative explanation is that exotherm peak of IME-8 is lower than expected because considerable reaction has already occurred at room temperature. In studies of the behavior of this resin system in bulk processing to yield resin discs and composites suggests that the latter explanation accounts for the DSC behavior. Processing characteristics to fabricate resin specimens are discussed in section 2.4.1.

The DSC behavior of the IME resins can be categorized into three groups. The first group, IME-1 and -5 showing low initial cure and low exotherm peaks (120°C), the second group IME-2, -3, -4, -6, -7, showing higher initial cure and higher exotherm peaks (200°C), and a third group resin C and IME-9 exhibiting the first exotherm peaks midway between the first and second group of resins. The DSC study suggests that for each system cure occurs at the initial starting temperature of the first exotherm and that a cure cycle for each system must consider this initial temperature.

### 2.3.3 Resin Gel Characteristics

The gel characteristics of the control resin and the eight IME resins were determined by observing the changes in physical appearance which occur while heating the powder (except for resin C) at two temperatures, 122°C and 150°C for various time periods. The results are summarized in Table 6.

#### 2.3.3.1 122°C Results

Resin C did not gel up to 1 hr at 122°C, indicating that a temperature higher than 122°C is required for curing of this resin system. Resins IME-1, -2, -3, -4 and -7 gelled rapidly within 4 to 13 min time range and IME-5 required 60 min for gelation at 122°C. IME-8 showed no melting at this temperature. The IR spectra of these two resins indicated that these resins were partially cured during the process of evaporating the MEK solvent used in preparing the homogeneous resin mixtures.

#### 2.3.3.2 150°C Results

At 150°C, resin C and IME-5 showed convenient gel times of 41 and 25 min, respectively. This temperature may be used for initial cure of those two resin systems. This temperature, however, is not suitable for curing IME-1, -2, -3, -4 and -7 resin systems, since their gel times ranging from 2 to 8 min are too short for easy resin processing. It appears from these studies (at 122° and 150°C) that a lower initial process temperature is required for resin systems IME-1, -2, -3, -4, and -8, perhaps closer to 100°C.

2.3.3.3 Under Contact Pressure

Resin specimens were placed in a closed mold at contact pressure, the temperature was raised from RT to the temperatures indicated in Table 6, and held at this temperature under contact pressure for the time periods indicated. This yielded an approximate gelation temperature/time cycle at contact pressure for each resin.

2.3.4 Melt Behavior of IME Resins

The capillary tube melt behavior of the IME-1 through -8 resin systems is listed in Table 7. Attempts to prepare resin specimens by heating the powder to the temperatures indicated resulted in sintered, porous solids, instead of glassy, clear solids observed in the melting point capillary. Apparently, in a mold poor heat transfer through the powder and cure of the outer surface of the powder prevents melting and consolidation. Resin specimens were prepared by fast heat-up under contact pressure, as described in section 2.4.1.

2.3.5 Investigation of Cure Cycles

Based on gel characteristics, DSC characteristics and melt behavior, several cure cycles incorporating pressure were investigated in attempts to prepare neat resin specimens (2.54 cm diameter x ~.63 cm thick) for basic physical and thermal property measurements. Table 8 summarizes the cure cycles investigated.

In each cure cycle, the solventless and homogeneous uncured powdered resin mixtures were treated at the initial temperature indicated (122°C or 177°C) based on gelation experiments (Table 6), by placing the mold containing the powder in a preheated press and immediately applying 0.69 MPa to consolidate the specimens. Pressure was maintained under these conditions for 1 hr, followed by a temperature increase as noted in Table 8. Temperature and pressure were maintained for the time periods indicated. The rate of temperature rise between temperatures was 2-3°C/min. Each resin responded differently to the treatment, yielding in most cases glassy specimens with considerable voids or sintered-like specimens.

Resin C is a liquid under these conditions allowing resin specimens to be cured in molds without pressure. For IME-1 and -5, pressure caused squeeze-out of the resins from the mold, indicating that these two systems are viscous liquids near gelation. IME-2, -3, -4, and -7 yielded porous specimens. Resins IME-4, -7, and -8 appeared to be almost completely cured even before exposure to any of the cure cycles. This was indicated by the solubility test and the IR spectra of the solventless resin mixtures. More evidence for this is that even at high pressure 3.45 MPa and high temperature (210°C), no melting or consolidation of the resin occurred. Considerable advancement of these resins must occur during solvent evaporation at 60°C in vacuum. Except in the cases of C, IME-1 and IME-5, the use of pressure was identified to be essential in forming consolidated resin specimens.

The DSC thermograms of each resin specimen cured by the various cure cycles showed that in all cases, cycles 2, 4 and 6 which include additional cure of each resin at 204°C for 24 hrs over the initial lower temperature cycle (cycles 1, 3 and 5) increase the initial softening temperature of the resin by 20° to 40°C depending on the system. Moreover, for the cure cycles which included a 24 hr postcure, the endotherm range did not vary significantly.

Based on these experiments, it was found that preparation of IME powders of resins IME-4, -6, -7, and -8 for resin specimen fabrication must be carried out at room temperature within a 2-3 hr period. Higher temperatures and longer times cause "B" staging, or advancement of the resin, which prevents consolidation of the powder into void free specimens. For the other resin systems, powder can be prepared by solvent evaporation at room temperature followed by vacuum treatment at 70°C for 2 hrs. Although data from capillary melt behavior and open mold gel characteristics of resin powders provide a guide to resin specimen preparation, or even composite fabrication, the data do not always correlate with fabrication of resin specimens from powder. As mentioned in section 2.3.4, this is most likely due to thermal lag experienced by bulk powder in a metal mold relative to small quantities of powder in capillary tubes or thin walled aluminum cups. Based on these cure cycle studies, the recommended cure cycle to prevent formation of voids for preparation of resin specimens involved fast heat up to the point where flow is observed, followed by the application of pressure at the gel point.

## 2.4 Resin Characterization

### 2.4.1 Disk Fabrication

For the resin systems under study, only the control resin is capable of being fabricated into neat resin specimens by pouring the liquid resin mix at 150°C into preheated (150°C) molds. All the other resin systems are solid mixtures at room temperature and efforts to form liquids as one raises the temperature, either slowly or rapidly, in vacuum or at ambient pressure to some softening or melt temperature results in a cured sintered-like specimen or a voidy glassy specimen. As a result, ASTM tensile specimens could be fabricated only for resin C. Miniature tensile specimens were fabricated for IME-1 and IME-5 resin systems by vacuum processing techniques described below. Neat resin disks 2.54 cm diameter x 0.48 cm to 0.635 cm thick samples of IME-1 through -8 were fabricated by compression molding techniques or by vacuum heat treatment starting with solvent-free resin powder. The conditions for preparing these specimens are listed in Table 9.

### 2.4.2 Physical Properties of Cured IME resins

#### 2.4.2.1 Density, Coefficient of Thermal Expansion and Cure Shrinkage

The density, coefficient of thermal expansion, and shrinkage due to cure of each IME resin are listed in Table 10. The IME-1 through -8 resins exhibit higher densities than typical epoxies due to the 6F-diamine curing agent. The shrinkage and coefficient of thermal expansion values are typical of epoxy resins.

#### 2.4.2.2 Resin Moisture Absorption

Small sections (0.64 cm section of a 2.54 cm diameter specimen) of each resin system were subjected to three conditions of moisture: distilled water at RT for 24 hrs, 87% RH, 82°C to saturation (approximately eight days) and boiling distilled water for 72 hrs. The results of the moisture gained due to these exposures are listed in Table 11. A comparison of the moisture absorbed by the IME resins for all three conditions with the moisture absorbed by a typical epoxy under the same conditions shows that the IME resins absorb considerably less moisture.

### 2.4.3 Resin Thermal Properties of Epoxy Resins

#### 2.4.3.1 Differential Scanning Calorimetry (DSC), Thermomechanical Analyses (TMA) and Thermogravimetric Analysis (TGA)

DSC, TMA and TGA thermograms of the IME resin are shown in Figs. 17-19. Data derived from the DSC, TMA and TGA thermograms of the IME resins are listed in Table 12. Where they appear, the first and second peaks of the DSC endotherms are listed for each resin. The second peak approaches the glass transition temperature of the resin as defined by TMA, but the correlation between the second DSC endotherm peak (Tg) and the glass transition temperature (Tg) of the resin is poor. The weight percent loss of each resin system at 300°C and 800°C (in air) based on TGA data is listed in Table 12. The weight loss experienced by the IME resins is considerably less than a typical epoxy.

#### 2.4.3.2 Resin Char Yields

The resin char yields were determined by two techniques; (1) exposure in a muffle furnace at 800°C for 3 min, and (2) TGA weight loss up to a temperature of 800°C in air. The data listed in Table 13 shows that the char yields of the IME resin are considerably greater than the control resin and a typical epoxy.

2.4.4 Resin Mechanical Properties

2.4.4.1 Tensile Properties

Because of the difficulties encountered in fabricating resin specimens from the IME resin systems, miniature resin systems 5.08 cm long x 0.635 cm wide, having a 0.32 cm reduced section in the 1.27 cm gage length were fabricated for IME-1 and -5. Standard ASTM resin specimens were prepared for the IME-1 control resin, C. The results of the tensile properties are listed in Table 14. Resin system IME-1 containing 6F-DDS has a higher strain at failure than the control resin with DDS or IME-5 with 6F-DDS and DDS.

2.4.4.2 Compression Strength

For resin samples IME-2, -3, -4, -6, -7, and -8 small samples of resin were cut from the 2.54 cm diameter disks for compression measurements. These specimens measured 1.90 cm long x 0.635 cm x 0.48 cm to 0.635 cm. Similar size specimens were cut for resin C and IME-1 and -5. The results of the compression strengths are listed in Table 15. The compression strengths of the IME resins compare favorably with a typical epoxy and with the control resin. The high compressive strength of the IME-8 resin relative to the other resins is noted.

2.5 Characterization of Celion 6000/IME Epoxy Resin Composites

2.5.1 Fabrication of Prepreg

Prepregs were fabricated by drum winding epoxy sized Celion 6000 graphite fibers and brush application of an acetone solution of the resin to the dried fiber, calculated to yield a composite with a fiber volume of 60 $\pm$ 2%. The prepreg tapes were air dried on the drum at room temperature, then vacuum dried at 70°C for 2 hrs to remove traces of acetone, except prepregs containing IME-4, 6, 7 and 8. These were dried at room temperature in vacuum for 2 hrs, instead of 70°C to prevent advancement of the resin. The plies were stacked unidirectionally and processed by autoclave or compression molding techniques, as described below.

2.5.2 Processing Parameters for Celion 6000 Epoxy Resin Composites

2.5.2.1 Autoclave Processing Studies

The prepregs were cut 15.24 cm x 20.32 cm and the plies were placed on an aluminum plate previously sprayed with Teflon release agent, and the vacuum tube was placed in position. The stacked plies were surrounded by 1.27 cm width of 0.635 cm thick felt. A Teflon release ply followed by a glass bleeder cloth were placed on top of the stacked plies, and finally the system was made vacuum tight in a Kapton bag. The autoclave process parameters utilized for each autoclaved process composite are listed in Table 16. Composites were postcured at 204°C for 3 or 24 hrs after some properties were measured in the as-fabricated condition.

#### 2.5.2.2 Compression Molding Processing Studies

A series of 10 ply 10.1 cm x 15.24 cm x 0.254 cm composites were fabricated by compression molding techniques to develop processing parameters for fabrication of larger composites. An open ended mold was used to follow the gelation point by probing the laminate as a function of time at specific temperatures. This information is required to produce void free compression molded composites. Of critical importance in the fabrication process is the time/temperature parameter for the application of pressure in the initial stages of gelation. Based on the experience with these composites, another series of larger composites (15.24 cm x 25.4 cm x 0.254 cm) were fabricated to a fixed volume using stops on the mold to control composite thickness and fiber content. The composites fabricated and the conditions used in the fabrication process are listed in Table 17. These composites were molded to a constant volume for better control of the final composition (fiber 60 vol %, resin 40 vol %).

### 2.5.3 Physical Properties of Celion 6000 Epoxy Resin Composite

#### 2.5.3.1 Composition, Density and Ply Thickness

The density, theoretical density, experimental resin, fiber and void volume and composite ply thickness are listed in Table 18 for the autoclave molded 10 ply composites (15.24 cm x 25.4 cm x ~.254 cm). This calculation assumes zero void. For a 70 fiber vol % composite, a void content of 2% can cause a density decrease of 0.02 g/cc, while for a 60 fiber vol % composite, a void content of 2% can decrease the density by 0.05 g/cc. Of significance in the data in Table 18 is the high void content (2 to 5%) and low composite ply thicknesses, in mils/ply. Both high void and low ply thickness (high fiber content) tend to lower the shear strength. The density, calculated resin and fiber volume percents, experimental resin, fiber and void volume percents and composite ply thickness for the compression molded 9 ply composites (15.24 cm x 25.4 cm x ~0.254 cm) are listed in Table 19. In this series the fiber and resin weight before processing were determined, and the final composite weight after removal of excess resin flash was determined to calculate a resin and fiber volume, assuming zero void. The calculated and experimental values are within experimental error.

### 2.5.4 Moisture Absorption Properties of Celion 6000 Epoxy Resin Composites

The autoclave and compression molded fabricated composites were subjected to three conditions of moisture: immersion in distilled water at room temperature for 24 hrs, 87% RH at 82°C to saturation (approximately 8 days), and boiling distilled water for 72 hrs. The results of these moisture absorption studies are listed in Table 20 for each moisture condition. Flexure specimens were used to determine moisture absorption for each condition. To determine size effects in moisture absorption shear specimens were exposed to the 87% RH, 82°C condition to compare with the flexure specimens. In general, the percent moisture absorption is greater for the smaller specimen because the surface to volume ratio is much greater in the shear specimen than in the flexure specimen.

### 2.5.5 Composite Thermal Properties

#### 2.5.5.1 Thermomechanical Analysis (TMA) and Thermogravimetric Analysis (TGA)

TMA and TGA thermograms of the Celion 6000 composite series are shown in Figs. 20-21 respectively. The glass transition temperatures (Tg) of each composite system as determined by TMA analysis, and the weight losses at 300°C and 800°C by the TGA method are listed in Table 21. A comparison of the Tg's derived from composites (Table 21) with Tg's derived from resin specimens (Table 12) shows that the Tg's derived on composites compare favorably with those derived from neat resins. The agreement between the two types of specimens (autoclave and compression molded) is fairly good. The presence of a first transition in a composite containing a particular IME resin and the absence of a first transition of another composite containing the same IME resin is not understood. Also, the variation of Tg's between different composites of the same resin is not understood.

#### 2.5.5.2 Char Forming Properties and Fiber Containment Characteristics

Two techniques were utilized to determine the char forming characteristics of the Celion 6000 epoxy resin composites. The first technique utilized the TGA method, whereby the weight loss as a function of temperature was determined up to a temperature of 800°C in air or higher. In the second technique, the char yield was determined after a 3 min exposure in a muffle furnace at 800°C. The results of the tests listed in Table 22 show that about all IME systems exhibit good char forming capabilities, with IME-1 and -3 exhibiting better char yield than the other resin systems. For the composites investigated, the fiber containment yield of the composites is essentially 100%, showing that the char encapsulates the fiber, preventing release to the atmosphere.

### 2.5.6 Microscopic and Ultrasonic Examination of Celion 6000 Epoxy Resin Composites

Polished transverse cross sections of composites 33-C, 20-IME-1, 34-IME-1, 32-IME-2, 28-IME-3, 23-IME-4, 29-IME-4, 24-IME-5, 30-IME-5, 25-IME-6, 31-IME-6, 26-IME-7 and 27-IME-8 were examined by optical microscopy. Composite quality in terms of fiber distribution, fiber content and void content was assessed. Optical micrographs of each composite system at 15X and 200X were prepared. Except for composites 20-IME-1, 23-IME-4, 25-IME-6 which showed a considerable quantity of surface voids, the quality of all composites with respect to voids, fiber content and fiber distribution is excellent. Optical micrographs of the above mentioned composites are shown in Figs. 22a-m.

Ultrasonic "C" scan of the above mentioned composites was performed at a frequency of 3.5 MHz. The "C" scan traces of these composites agreed with the optical micrograph examinations. The "C" scan traces of composites 20-IME-1, 23-IME-4 and 25-IME-6 revealed the presence of considerable defects, attributed to voids. The "C" scan traces of the remaining composites suggested that these composites were free of defects or voids, in agreement with optical microscopy and void content determination.

### 2.5.7 Mechanical Properties of Celion 6000 Epoxy Resin Composites

#### 2.5.7.1 Interlaminar Shear Properties

The interlaminar shear strength (ILSS) properties of each composite system in the dry state are listed in Tables 23, 24, 26, 27, and 29 while the ILSS properties after moisture exposure are listed in Tables 25, 28, and 30. ILSS data for autoclave molded composites is represented in Tables 23, 24, and 25, while ILSS data for compression molded composites is listed in Tables 28 through 32. In general, except for composites which contain visible surface defects or internal defects (20-IME-1, 30-IME-5, 38-IME-8, 23-IME-4) the range of dry ILSS strength at RT (82.4 MPa to 103 MPa), at 150°C (41.2 to 61.8 MPa) and at 177°C (34.3 to 54.9 MPa) is considered acceptable for novel systems. These values are equivalent to state-of-the-art Celion 6000/epoxy resin composite systems. Another generalization is that the shear strengths of autoclave molded and compression molded composites are approximately equivalent. Postcuring (3 hrs or 24 hrs) has no effect on RT ILSS properties, as expected (see Table 25). However, a 24 hr postcure at 204°C caused considerable improvement in the dry 150°C and 177°C shear strength as can be noted by comparing data in Table 29 with data in Table 27. Therefore, postcuring is necessary in order for these composite systems to have a 177°C temperature capability. The effect of moisture on RT and elevated temperature shear properties can be seen by comparing dry strength (Tables 24, 27, 29) with wet strength (Tables 25, 28 and 30). Composite systems with resins IME-1 and IME-5 exhibited the best wet strength retention of all the composite systems investigated.

### 2.5.7.2 Flexural Properties

The flexural properties of each composite system in the dry state are listed in Tables 31, 32, 34, 35, 37 and 38, while the same properties after moisture exposure are listed in Tables 33, 36, and 39. In general, compared to state-of-the-art epoxies, the RT flexural properties are considered acceptable. However, only composites containing the novel resins IME-1 and IME-5 exhibited acceptable 150°C and 177°C flexural strengths relative to state-of-the-art Celion 6000/epoxy resin composites. However, it should be cautioned that the composites listed in Tables 32, 33 and 34 failed in compression, suggesting that the resin was yielding. A more brittle resin would cause the composites to fail in tension or shear. Except for composites which contain visible or internal defects (20-IME-1, 23-IME-4, 30-IME-5, 38-IME-8), the flexural strengths exhibited by the compression molded composites (Table 38) are superior to the flexural strengths exhibited by the autoclave molded composites (Table 32). Postcuring of the composites for 24 hrs has no apparent effect on the RT flexural properties, but caused a large increase in the 177°C flexural strengths, as noted by comparing Table 35 with Table 38.

The effect of moisture on the flexural properties at RT and elevated temperatures can be determined by comparing the dry flexural strength data (Tables 32, 35 and 38) with the wet flexural data for the corresponding composites (Tables 33, 36, 39). Similar to what was found in the study on the effect of moisture on ILSS, examination of the flexural strength data reveals that composites containing novel resins IME-1 and -5 exhibited greatest resistance to degradation of RT, 150°C and 177°C flexural strength after exposure to moisture.

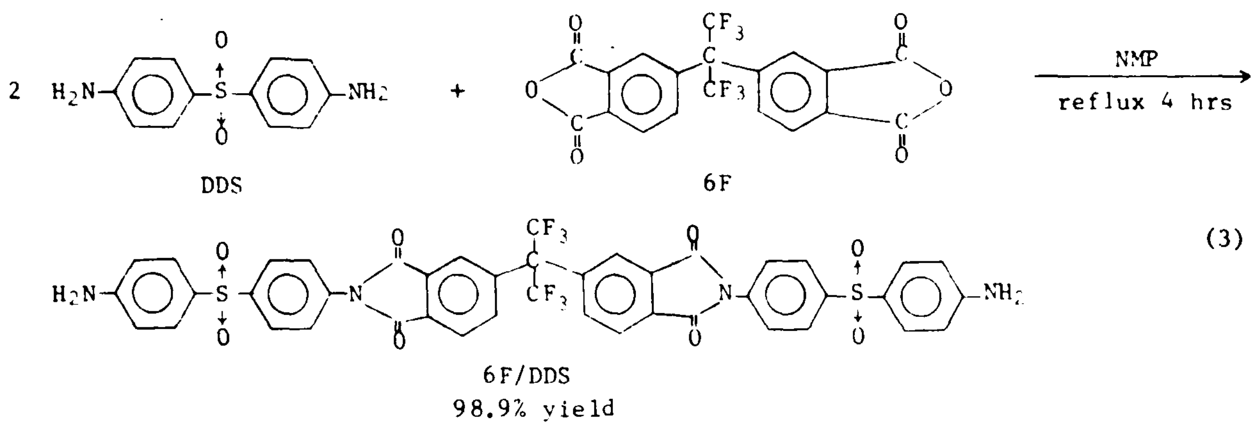
## 3. EXPERIMENTAL

### 3.1 Synthesis of Bisimide Amines (BIA's)

The synthesis, purification, and characterization of the four required bisimide amine (BIA's) hardeners, are described below.

#### 3.1.1 4,4'-[2,2,2-Trifluoro-1-(trifluoromethyl)ethylidene]bis[N-(p-sulfanilylphenyl)phthalimide],(6F-DDS)

The synthetic reaction for this compound is depicted in Equation (3).



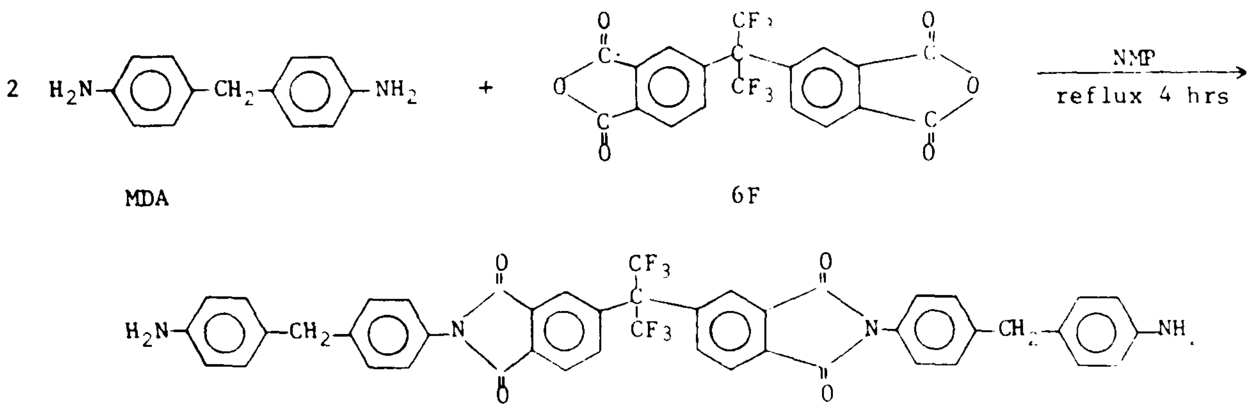

(3)

To a stirred solution of DDS (0.50 mole, 124.2g) under refluxing conditions in 350 ml of NMP, a solution of 6F (0.25 mole, 113.6g) in 350 ml NMP was added over 1/2 hr period. The reaction mixture was stirred and refluxed for 4 hrs. The solution was then concentrated under vacuum (0.5 mm Hg) to half its initial volume, and allowed to cool to room temperature. The solution was poured, while stirring into an ice-water mixture (350g-350g). The tan solid material which precipitated was filtered and washed several times with 100 ml of distilled water. After drying in vacuum at 60°C overnight, a crude yellow solid was obtained. It was purified by dissolving the solid in hot acetone, and after cooling to room temperature, precipitated by the addition of cold distilled water. The precipitate was collected by suction filtration and washed several times with distilled water until the filtrate appeared clear and colorless. A yield of 198g (91% yield) of pale yellow solid was obtained. The purity was estimated to be 90% as determined by GPC (Fig. 1); IR (chloroform solution) (Fig. 3); 3495 (w, amine N-H), 3400 (m, amine N-H), 1780 (m, imide C=O), 1725 (s, imide C=O), 1300 (s, sulfone S=O), 1230 (bs, sulfone S=O), 1150 (s, sulfone S=O); NMR ($D_6$ acetone) (Fig. 4): $\delta$, 2.75 (2H, $H_2O$), 2.1 (6H, acetone), 5.15 (4H, $NH_2$), 6.6-6.9 (m, H aromatic H), 7.50-8.35 (m, 16 H, aromatic H). Endotherms at 105°C and 240°C as determined by DSC (Fig. 5).

Calc. for $C_{43}H_{26}N_4F_6S_2O_8$ (MW 904.8): C, 57.08; H, 2.88; N, 6.20; F, 12.61; S, 7.08
Found: C, 56.58; H, 3.33; N, 6.14; F, 12.86; S, 6.99.

3.1.2 4,4'-[2,2,2-Trifluoro-1-(trifluoromethyl)ethylidene]bis[N-[τ-(p-aminophenyl)-p-tolyl]phthalimide],(6F-MDA)

This compound is prepared according to Equation (4).




(4)

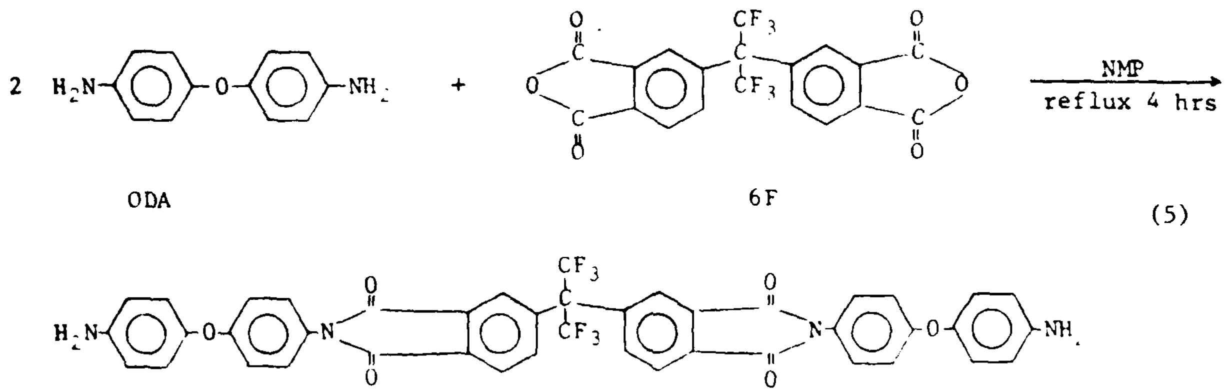
6F/MDA
99.6% yield

To a stirred solution of MDA (95.2g, 0.48 mole) in 350 ml of NMP, a solution of 6F (108g, 0.24 mole) in 350 ml of NMP was added over 1/2 hr period. The reaction mixture was stirred and refluxed for 4 hrs. The solution was concentrated under vacuum (0.5 mm Hg) to half of its initial volume. Upon cooling to room temperature, the concentrated solution was poured into ice-water mixture. The brown solid material which precipitated was filtered and washed seven times with 100 ml distilled water. After drying in vacuum oven at 60°C overnight, this yielded crude brown product (192.4g, 99.6% yield). The crude product was dissolved in a minimum quantity of hot acetone. After cooling to room temperature, cold distilled water was added to the acetone solution. The precipitates were collected by suction filtration and washed several times with distilled water until the filtrate appeared clear and colorless. Drying of the recrystallized product gave an analytical sample of 6F/MDA (154.5g, 80% yield): purity 99% as determined by GPC (Fig. 1); IR (chloroform solution) (Fig. 3); 2485 (w, amine N-H), 3380 (m, amine N-H), 1780 (m, imide C=0), 1720 (s, imide C=0). NMR ($CDCl_3$) (Fig. 4); $\delta$ 3.2 (s, 2H methylene group), 3δ-42 (m, 4H, $NH_2$), 6.4-7.5 (m, 16H, aromatic H), 8.0 (s, 6H, aromatic H), signals at $\delta$ 1.3-2.9 are impurity peaks. Melting point 230°C as determined by DSC (Fig. 5).

Calc. for $C_{45}H_{30}N_4F_6O_4$ (MW 804.7): C, 67.16; H, 3.73; N, 6.97; F, 14.18
Found: C, 66.50; H, 4.00; N, 7.09; F. 14.51.

3.1.3 4,4'-[2,2,2-Trifluoro-1-(trifluoromethyl)ethylidene]bis[N-[p-(p-aminophenyoxy)phenyl]phthalimide](6F-ODA)

This compound was prepared in accordance with Equation (5).




(5)

6F/ODA
98.0% yield

A solution of ODA (196.23g, 0.98 mole) in 400 ml of NMP was added to a 2000-ml four-necked round-bottom flask equipped with a stirrer, a reflux condenser, an addition funnel, and a thermometer. A solution of 6F (216.0g, 0.49 mole) in 600 ml of NMP was (dropwise) added over a 1 hr period to the ODA solution which was stirred and held at reflux. The progress of the reaction was followed by measuring the optical densities of the 6F anhydride band at 1840 $cm^{-1}$ and the 6F/ODA imide band at 1720 $cm^{-1}$. The reaction was essentially complete in 4 hrs refluxing time. After 4 hrs, the reaction solution was concentrated under reduced pressure (2 mm Hg) to half of its original volume. Upon cooling to room temperature, the concentrated solution was poured into 1000 ml distilled water to precipitate yellowish products. The fine crystalline materials were suction filtered, washed with distilled water, and dried in a vacuum oven at 60°C overnight to give 388g (98% yield) of crude products. The crude products were recrystallized from acetone-water to afford 356.3g (90% yield) off-white recrystallized products. The GPC chromatogram of the recrystallized materials (Fig. 2) revealed two major peaks (99%) and traces of starting amine indicating a compound of good purity. The IR, NMR, DSC, and elemental analysis of the recrystallized products were determined and are presented as follows:

IR ($CDCl_3$) (Fig. 3):

3480 (w, amine N-H), 3370 (m, amine N-H), 1780 (m, imide C=O), 1720 (S, imide C=O), 1220 (bs, ether C-O).

NMR ($CDCl_3$) (Fig. 4): $\delta$ 3.50 (bs, 4H, $NH_2$), 6.50-7.60 (m, 16H aromatic H), 7.8-8.2 (m, 6H, aromatic H). Note that the signals in the region of $\delta$ 2.20 to 3.00 are probably due to solvents and impurities.

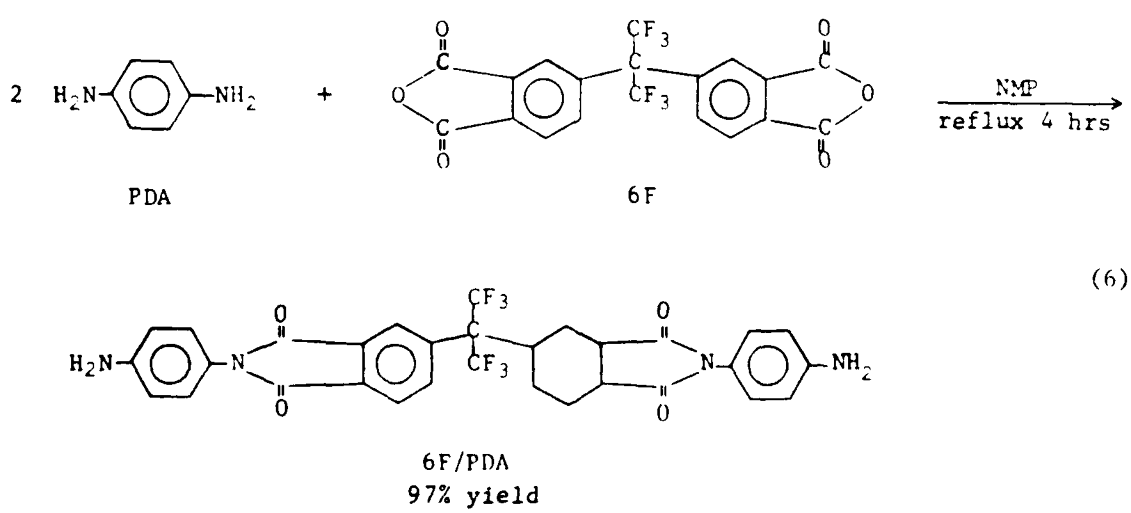
DSC (Fig. 5): Endotherms at 90, 165, 215, and 325°C.

Elemental Analysis:

Calc. for $C_{43}H_{26}N_4F_6O_6$, (MW 808.6):
C, 63.86; H, 3.22; N, 6.93; F, 14.11

Found: C, 64.27; H, 3.86; N, 6.69; F, 13.80

3.1.4 4,4'-[2,2,2-Trifluoro-1-(trifluoromethyl)ethylidene]bis[N-(p-aminophenyl)phthalimide](6F-PDA)




(6)

The synthetic reaction of this compound is shown in Eq. (6). The detailed synthetic procedure followed the same method as described in Section 3.1.2 for 6F-MDA. To a stirred and refluxing solution of PDA (81.2g, 0.75 mole) in 350 ml of NMP, a solution of 6F (165.2g, 0.37 mole) in 700 ml of NMP was rapidly added dropwise. After work-up followed by recrystallization from acetone-water, this reaction afforded 224g (97% yield) of 6F/PDA in dark purple color:

Purity 99% as determined by GPC (Fig. 2).

NMR ($D_6$ acetone) (Fig. 4): $\delta$ 3.8 (bs, 4H, $NH_2$), 6.7-7.6 (m., 8H, aromatic H), 7.90-8.3 (d, 6H, aromatic H). Note that the other signals between 0.7 to 2.2 are probably due to solvents and impurities.

M.P. 330°C as determined by DSC (Fig. 5).

Elemental Analysis:

Calc. for $C_{31}H_{18}N_4F_6O_4$, (MW 624.5),
C, 59.62; H, 2.88; N, 8.97; F, 18.27

Found: C, 59.79; H, 3.26; N, 8.57; F, 17.99

## 3.2 *Preparation of Homogeneous* Resin Powders

Except for the base resin system MY 720 (DDS) (IME-1), homogeneous resin powders were prepared by dissolving the epoxy resin MY 720 and curing agents in acetone, and concentrating the mixture in a hood at room temperature to a solid powder. Because of the high reactivity of imide epoxies, residual solvent was removed at room temperature in vacuum over a 2 hr period. The homogeneous powders prepared in this manner were used for the DSC, TGA, and resin fabrication studies. The stoichiometric quantities of each component used in these studies and in the infrared spectroscopy studies are listed in Table 3.

## 3.3 Resin Cure and Fabrication Studies

### 3.3.1 Cure Studies

The techniques utilized and properties measured to determine the cure cycle of each IME resin system were infrared spectroscopy (IR), differential scanning calorimetry (DSC), gelation temperature/time behavior, and melt behavior. A Perkin-Elmer Model 451 infrared spectrometer was used in the infrared studies. Films of each resin system were deposited on a sodium chloride salt plate by concentration of a solution of the resin in methylethylketone. The film was treated for certain temperature/time sequences listed in Table 4. After each cure treatment, an infrared spectrum was taken. The thermal behavior of each uncured resin powder was followed by DSC, gelation experiments, and melt behavior. A DuPont 990 thermal analyzer attached to DuPont differential scanning calorimeter was used to make the DSC studies. A small quantity (5-10 mg) of powder was used for each DSC scan.

For the gelation temperature/time experiments, with and without pressure, a small quantity (~0.5g) of powder was placed in a thin walled aluminum dish, and placed on a hot plate already set at 122°C or 150°C. The time required for the powder to gel at 122°C or 150°C by probing and observing the flow behavior was determined. Similar experiments were carried out in a press preheated to 10°C, except contact pressure was applied to the powder immediately after it was placed in the press.

### 3.3.2 Fabrication Studies

Tensile specimens of the control resin were prepared by pouring a solution of the MY 720/DDS system in open molds (ASTM D638-68, Type I size) and subjecting the specimens to the cure cycle listed in Table 9. For the other resins, a compression molding technique was utilized. The resin powder (5.0g) was placed into a 2.54 cm diameter mold. The assembly was placed into a preheated press, a pressure of 6.89 MPa was applied to consolidate the powder. Pressure was released, and the specimen was then compression molded according to the details in Table 9. In addition to 2.54 cm diameter resin disks, 3.81 cm x 5.08 cm x 0.635 cm resin specimens of IME-1 and -5 were prepared, also according to the details in Table 9.

## 3.4 Characterization of IME Epoxy Resins

### 3.4.1 Physical Properties

Density was determined by measuring the volume of a symmetrical sample, and then determining the weight of the sample. The density was calculated as follows: density = weight in grams/volume (cc) = g/cc. The cure shrinkage of each resin system was determined as follows: % shrinkage = diameter of mold (RT) - diameter of resin spec (RT)/diameter of mold (RT). The coefficient of thermal expansions were determined on 0.47 cm x 0.47 cm to 0.64 cm x 1.90 cm x 2.54 cm length specimens cut from the 1" diameter disks by dilatometric techniques. A Theta Industries Dilatronic II Research Model Dilatometer was used.

### 3.4.2 Thermal Properties

A DuPont 990 thermoanalyzer equipped with a 943 thermomechanical analyzer (TMA) and a DuPont thermogravimetric analyzer (TGA) were used to determine the glass transition temperature (Tg) and weight losses in air as a function of temperature of each resin specimen. The DSC behavior was also determined on a DuPont 990 thermoanalyzer equipped with a DuPont differential scanning calorimeter (DSC).

The aerobic char yield of each resin sample was determined in air by the TGA technique up to a temperature of 800°C. A C&M high temperature muffle furnace was also used for determination of aerobic char yield. The sample was placed in the oven at 800°C for a period of three (3) minutes. The char yield was determined as follows: % resin char yield = wt char after burning X100/wt resin before burning.

### 3.4.3 Mechanical Properties

Tensile specimens of the control resin were prepared according to ASTM D638-68, Type I size, and measured according to ASTM D638-68 at a crosshead speed of 0.05 in/min. Miniature tensile specimens of IME-1 and -2 were cut from 3.81 cm x 5.08 cm x 0.635 cm coupons. Final dimensions of the test specimens were 5.08 cm long x .635 cm wide, having a 0.32 cm reduced section in the 1.27 cm wide gage section. The samples were tensile tested at a crosshead speed of 0.127 cm/min.

The samples used for coefficient of thermal expansion measurements were also used for the compression strength tests. These specimens measured 0.48 cm x 0.48 cm to 0.635 cm x 1.90 cm x 2.54 cm long, and were cut from the 2.54 cm diameter disks. Except for the sample size, the samples were tested according to ASTM D695-69 at a crosshead speed of 0.127 cm/min.

## 3.5 Characterization of Celion 6000 Epoxy Resin Composites

### 3.5.1 Celion 6000/IME Tape and Composite Fabrication

For each composite specimen fabricated, a calculated and measured quantity of epoxy resin was used to give a composite with 60 vol % fiber and 40 vol % resin. This was accomplished by applying , by brush, a solution of the resin in methylethylketone to a prewound 15.24 cm or 10.16 cm x 43.2 cm Celion 6000 dry tape. A 10% excess of resin was applied to account for loss in transfer and resin bleed-out during laminate fabrication. After application of the resin solution to the dry tape, the tape was allowed to stand at room temperature for 2 hrs. For C, 1, 2, 3, and 5 resin system, trace solvent was removed in vacuum at 60°C for 2 hrs. For IME-4, -6, -7, and -8 resin system trace solvent was removed at room temperature in vacuum for 2 hrs. The tape was cut in plies and stacked on a plate for autoclave processing or in a mold for the compression molding process. For the autoclave technique, the plies were stacked on a Teflon sprayed 30.5 cm x 30.5 cm x 0.64 cm aluminum plate. This was surrounded by wide bleeder felt. One sheet of a Teflon coated glass scrim cloth was placed on the top ply, followed by one sheet of heavy duty glass cloth fabric. Adjacent to one side of the plies a 0.635 cm ID copper tube was fixed to the felt with copper wire, extending about 20.32 cm into the composite assembly and 15.24 cm outside of the assembly. A thermocouple for temperature measurements was placed along side the copper tube extending into the laminate. Kapton polyimide film was placed over the assembly and sealed into position with elastomeric sealant tapes to provide a vacuum tight assembly. An aluminum plate was placed on top of the assembly. The system was evacuated at room temperature for an hour, and then placed in a preheated press.

### 3.5.2 Composite Characterization Techniques

#### 3.5.2.1 Physical Properties

The density of each composite was determined by the liquid displacement technique. Fiber, resin and void volume was determined by the acid digestion method. Optical micrographs were determined on polished cross section of 2.54 cm wide specimens.

#### 3.5.2.2 Description of Experimental Method for Ultrasonic C-Scans of Composite Panels

Ultrasonic C-scans were performed on the composite panels to determine their structural integrity and homogeneity. These tests were carried out in a water medium at a frequency of 3.5 MHz using a reflector plate technique shown in Fig. 23. The ultrasonic energy emitted by the transducer passes through the panel, is reflected off an aluminum plate, and then passes back through the panel where it is received by the same transducer. The amplitude of this received signal is proportional to the structural properties of the composite. Quantitative amplitude modulated C-scans were performed. The output of these tests is a 'hills and valleys' map of the ultrasonic signal as it varies from position to position on the panel. To insure that the C-scans are comparable from specimen to specimen, system gain and alignment are calibrated using an aluminum plate 0.32 cm thick before running each composite panel.

#### 3.5.2.3 Thermal Properties

The thermal properties of composites were determined by the same procedures used on the resins, described in section 3.3.2. The char yields were determined by the technique described in 3.3.2. The following calculation was used for composites charred in the muffle furnace.

$$\% \text{ resin char yield} = \frac{\text{Wt char after burning}}{\text{Wt resin before burning}} \text{ X100}$$

The weight of resin before burning char and after burning was determined by $H_2SO_4$/ peroxide digestion of composites before and after burning. The fiber containment measurement was carried out on a muffle furnace charred composite specimen immediately after it was removed from the 800°C furnace. The specimen was impacted with a 100 gm weight which was dropped from a height of 8 cm. The weight of the debris was determined. The composition of the composite (fiber weight) was also determined on another sample by the $H_2SO_4$/peroxide method before and after burning. The fiber containment was calculated as follows:

$$\% \text{ fiber containment} = \frac{\text{Wt fiber after burning}}{\text{Wt fiber before burning}} \text{ X100}$$

#### 3.5.2.4 Mechanical Properties

Interlaminar Shear Strength Test - The short beam shear test was conducted at a span-to-depth ratio of 4:1. The loading nose is 0.635 cm diameter and the support pins are 0.48 cm diameter. Crosshead speed was 0.127 cm/min. Nominal specimen dimensions are 0.635 cm wide x 1.27 cm long x .254 cm thick.

Flexural Test - A three point load bend test at a span-to-depth ratio of 20:1 was conducted on composite specimens. Crosshead speed was .127 cm/min. Specimen dimensions are nominally 0.635 cm wide x 5.59 cm long x .254 cm thick.

# 4. CONCLUSIONS

1. Each BIA containing resin exhibited twice the char yield of the control resin MY 720/DDS (40% char vs 20% char).

2. The moisture absorption properties of these bisimide amine cured epoxies (IME's) were considerably less than state-of-the-art epoxies (0.5 wt % versus 2.0% saturation at 87% RH at 315 K (82°C)).

3. The strain-to-failure of the MY 720/DDS (control resin, C) resin system was improved 25% by replacement of DDS and 6F-DDS (MY 720/6F-DDS) (1.25% versus 0.99%).

4. Two of the composite systems Celion 6000/IME-1 and Celion 6000/IME-5 emerged as having superior properties relative to the other Celion 6000/IME composite systems and also relative to state-of-the-art graphite epoxy systems. This was demonstrated by excellent values for the wet shear and flexural strengths and moduli at 150°C and 177°C. For example, the 150°C and 177°C wet shear strengths of these systems are approximately 7000 psi and 5000 psi respectively. The 150°C and 177°C wet flexural strengths and moduli for these two systems are 1030 MPa, 107 GPa and 859 MPa, 101 GPa respectively.

5. Based on the resin properties, processing characteristics and composite properties, bisimide amine cured MY 720 epoxy systems MY 720/6F-DDS (IME-1) and MY 720/6F-DDS/DDS (IME-5) approach the objectives of the program to develop a resin with improved toughness, char yield and moisture resistance over state-of-the-art resins.

## 5. RECOMMENDATIONS

Additional studies with these novel bisimide amine cured epoxy systems should comprise methods to modify the two best bisimide amine cured MY 720 epoxy resins, MY 720/6F-DDS (IME-1) and MY 720/6F-DDS/DDS (IME-5) to improve the toughness characteristics of these resin systems.

## 6. REFERENCES


1. Serafini, T. T., Delvigs, P. and Vannucci, R. D., "High Char Imide Modified Epoxies", NASA TM 79226, also Vol. 12, Proc. of 12th National SAMPE Conf.

2. U.S. Patent 4,244,857, Serafini et al.

Table 1

Structures of Hardeners and Epoxy Resin

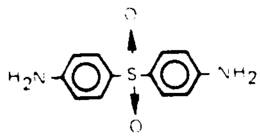
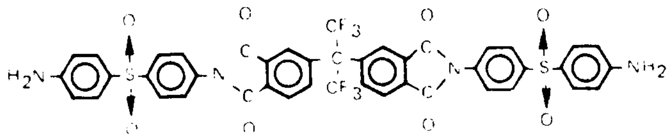
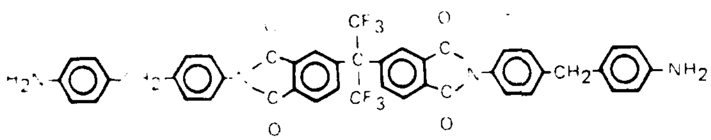
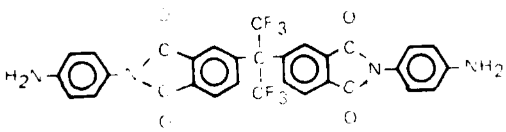
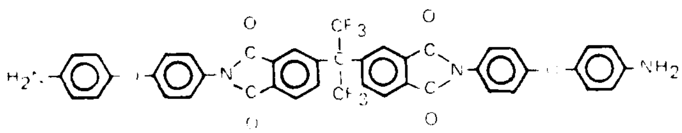
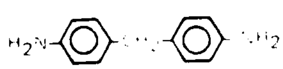
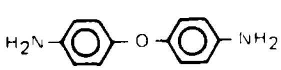
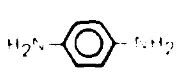
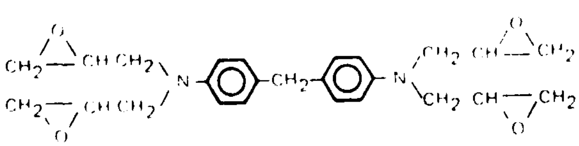
| Structure | Designation |
|---|---|
|  | DDS |
|  | 6F-DDS |
|  | 6F-MDA |
|  | 6F-PDA |
|  | 6F-ODA |
|  | MDA |
|  | ODA |
|  | PDA |
|  | MY 720 |

Table 2

Chemcial Compositions of Neat Resin Specimens

| Resin System No. | Resin System Epoxy/hardener(s) | Molecular Weight[1] Epoxy/hardener(s) | Mole Ratio of Epoxy/hardener(s) | Weight Ratios of Epoxy/hardener(s) g |
|---|---|---|---|---|
| C | MY 720/DDS | 500/248.4 | 0.012/0.012 | 6.00/3.00 |
| IME-1 | MY 720/6F-DDS | 500/904.8 | 0.0064/0.0064 | 3.20/5.79 |
| IME-2 | MY 720/6F-MDA | 500/804.7 | 0.0069/0.0069 | 3.45/5.52 |
| IME-3 | MY 720/6F-ODA | 500/808.6 | 0.0069/0/0069 | 3.45/5.58 |
| IME-4 | MY 720/6F-PDA | 500/624.5 | 0.008/0.008 | 4.00/4.99 |
| IME-5 | MY 720/6F-DDS/DDS | 500/904.8/248.4 | 0.0084/0.0042/0.0042 | 4.00/3.80/1.04 |
| IME-6 | MY 720/6F-MDA/MDA | 500/804.7/198.3 | 0.009/0.0045/0.0045 | 4.50/3.62/0.89 |
| IME-7 | MY 720/6F-ODA/ODA | 500/808.7/200.1 | 0.0088/0.0044/0.0044 | 4.40/3.56/0.88 |
| IME-8 | MY 720/6F-PDA/PDA | 500/624.5/108.1 | 0.0104/0.0052/0.0048 | 5.20/3.25/0.52 |

[1]Based on the chemcial formula of tetraglycidylmethylenedianiline (MY720), the molecular weight of MY720 should be 422. According to the manufacturer (Ciba Geigy), the average molecular weight of this epoxy resin is 500. The latter value is used throughout this investigation.

Table 3

Resin Systems Studied by IR

| Resin System No. | Resin Composition |
|---|---|
| C | 1.0 equiv. MY 720 + 1.0 equiv. DDS |
| IME-1 | 1.0 equiv. MY 720 + 1.0 equiv. 6F-DDS |
| IME-2 | 1.0 equiv. MY 720 + 1.0 equiv. 6F-MDA |
| IME-3 | 1.0 equiv. MY 720 + 1.0 equiv. 6F-ODA |
| IME-4 | 1.0 equiv. MY 720 + 1.0 equiv. 6F-PDA |
| IME-5 | 1.0 equiv. MY 720 + 0.5 equiv. 6F-DDS + 0.5 equiv. DDS |
| IME-6 | 1.0 equiv. MY 720 + 0.5 equiv. 6F-MDA + 0.5 equiv. MDA |
| IME-7 | 1.0 equiv. MY 720 + 0.5 equiv. 6F-ODA + 0.5 equiv. ODA |
| IME-8 | 1.0 equiv. MY 720 + 0.5 equiv. 6F-PDA + 0.5 equiv. PDA |

Table 4

Infrared Cure Study of Epoxy Resins by Following Epoxy/Aromatic Absorption Ratio

| Cure Cycle No. | Conditions | Resin System: C | IME-1 | IME-2 | IME-3 | IME-4 | IME-5 | IME-6 | IME-7 | IME-8 |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Absorbance Ratio 915 $cm^{-1}$/1512 $cm^{-1}$ | | | | | | | | |
| 1 | Room temperature vacuum, 1 hr | 0.181 | 0.184 | 0.123 | 0.101 | 0.136 | 0.280 | 0.088 | 0.087 | 0.142 |
| 2 | 1 hr at 122°C | 0.209 | 0.146 | 0.08 | 0.069 | 0.066 | 0.096 | 0.062 | 0.095 | 0.102 |
| 3 | 1 hr at 122°C + 1 hr at 177°C | 0.064 | 0.053 | 0.046 | 0.041 | 0.042 | 0.028 | 0.038 | 0.022 | 0.023 |
| 4 | 1 hr at 122°C + 2 hrs at 177°C | 0.054 | 0.028 | 0.025 | 0.023 | 0.023 | 0.022 | 0.042 | 0.019 | 0.032 |
| 5 | 1 hr at 122°C + 3 hrs at 177°C | 0.051 | 0.029 | 0.025 | 0.020 | 0.019 | 0.025 | 0.022 | 0.016 | 0.023 |
| 6 | 1 hr at 122°C + 4 hrs at 177°C | 0.037 | 0.029 | 0.024 | 0.016 | 0.021 | 0.022 | 0.017 | 0.011 | 0.019 |
| 7 | 1 hr at 122°C + 5 hrs at 177°C | 0.034 | 0.025 | 0.020 | 0.013 | 0.020 | 0.018 | 0.026 | 0.011 | 0.016 |
| 8 | 1 hr at 122°C + 5 hrs at 177°C + 1.5 hrs at 202°C | 0.021 | 0.027 | 0.0146 | 0.0116 | 0.018 | 0.014 | 0.027 | 0.003 | 0.0074 |
| 9 | 1 hr at 122°C + 5 hrs at 177°C + 2.5 hrs at 202°C | 0.021 | 0.0316 | 0.0135 | 0.0114 | 0.017 | 0.019 | 0.008 | 0.0016 | 0.0082 |
| 10 | 1 hr @ 122°C + 5 hrs @ 177°C + 24 hrs @ 202°C | 0.015 | 0.0316 | 0.0135 | 0.102 | 0.020 | - | - | - | - |

Table 5

DSC Data of Uncured Epoxy Resins

| Resin System | Temperature of Exotherm, °C | | | | | |
|---|---|---|---|---|---|---|
| | First Exotherm | | | Second Exotherm | | |
| | Starting Temp. | Peak Temp. | Final Temp. | Initial Temp. | Peak Temp. | Final Temp. |
| C | 138 | 155 | - | 155 | 225 | 275 |
| IME-1 | 100 | 120 | 150 | 170 | 240-270 Doublet | 285 |
| IME-2 | 145 | 215 | 245 | 250 | 285 | 300 |
| IME-3 | 145 | 210 | 250 | 255 | 285 | 310 |
| IME-4 | 140 | 230 | 260 | 260 | 275 | 290 |
| IME-5 | 100 | 120 | 135 | 140 | 220-235 Doublet | 250 |
| IME-6 | 135 | 195 | 245 | 245 | 280 | 290 |
| IME-7 | 135 | 195 | 245 | 245 | 285 | 295 |
| IME-8 | 120 | 155 | 200 | 200 | 240-265 Doublet | 285 |

Table 6

Gel Characteristics of Resins

| Resin System | Gel Time, min at 122°C | Gel Time, min at 150°C | Approx. Gelation Under Contact Pressure Temp. °C/min |
|---|---|---|---|
| C | liquid-like showed no gelation up to 1 hr | 41 | ~150/90 |
| IME-1 | 13 | 8 | 180/60 |
| IME-2 | 9 | 4 | 138-150/60 |
| IME-3 | 7 | 4 | 120-123/7 |
| IME-4 | 10 | 5 | 145-166/20 |
| IME-5 | 60 | 25 | 150/90 |
| IME-6 | no melting | no melting | 145/15 |
| IME-7 | 4 | 2 | 75-148/12 |
| IME-8 | no melting | no melting | ~146/10 |

Table 7

Capillary Tube Melt Behavior of Resin Powders

| Resin No. | Observations |
|---|---|
| IME-1 | Sinters 105, melts 110°C |
| IME-2 | Darkens, sinters 105°C, melts ~110°C |
| IME-3 | Melts 100-110°C<br>Gas evolution: 145°C |
| IME-4 | Melts 64°C |
| IME-5 | Melts 60°C |
| IME-6 | Melts 58-67°C |
| IME-7 | Melts 60-67°C<br>Gas evolution 170°C |
| IME-8 | Sinters 70°C, melts 88-130°C<br>Gas evolution: 165°C |

Table 8

Initial Cure Cycles Investigated for Fabrication of Epoxy Resin Specimens

| Cure Cycle No. | Process Conditions |
|---|---|
| 1 | RT → 122°C (2-3°C/min), at temp., apply 0.69 MPa, hold 2 hrs, 122°C→177°C (2-3°C/min), 0.69 MPa, hold 2 hrs |
| 2 | RT→122°C (2-3°C/min), at temp. 0.69 MPa, hold 2 hrs, 122°C→177°C (2-3°C/min), 100 psi, hold 2 hrs at 177°C, 177°C→204°C (2-3°C/min), hold 24 hrs, (atm pressure). |
| 3 | RT→150°C (2-3°C/min), at temp., 0.69 MPa, hold 1 hr, 150°C→177°C (2-3°C/min), 0.69 MPa, hold 2 hrs at 177°C. |
| 4 | RT→150°C (2-3°C/min), at temp. 0.69 MPa, hold 1 hr, 150°C→177°C (2-3°C/min), 0.69 MPa, hold 2 hrs, 177°C→204°C (2-3°C/min), hold 24 hrs, (atm pressure). |
| 5 | RT→150°C (2-3°C/min), at temp. 0.69 MPa, hold 1 hr, 150°C→201°C (2-3°C/min), 0.69 MPa, hold 2 hrs. |
| 6 | RT→150°C (2-3°C/min), at temp. 0.69 MPa, hold 1 hr, 150°C→201°C, (2-3°C/min), 0.69 MPa, hold 2 hrs, 201°C→204°C, hold 24 hrs (atm pressure). |
| 7 | RT→150°C (2-3°C/min) at temp. 0.69 MPa, hold 1 hr, 150°C→210°C (2-3°C/min), 0.69 MPa, hold 2 hrs. |

Table 9

Processing Parameters for Fabrication of Epoxy Resin Specimens

| Specimen No. | Initial Sample Preparation | Cure Cycle[1] |
|---|---|---|
| C | Mix MY720 + DDS, heat to 150°C, pour liquid in preheated (150°C) mold | 150°C/1 hr + 177°C/2 hrs + 204°C/2 hrs |
| IME-1 | Removed solvent at 70°C 2 hrs in vacuum to produce resin powder | 70°C vacuum, raise T to 150°C, hold ½ hr, raise T to 180°C/10 min, release vacuum, hold 180°C/2 hrs, raise T to 204°C, hold 204°C/2 hrs |
| IME-2 | Same process as IME-1 | Compression molded. Placed in preheated press at 180°C, consolidated sample at 0.69 MPa and removed pressure, flow occurs at 102°C, raise T to 150°C, gelation occurs immediately, applied pressure (0.69 MPa), raised T to 180°C, hold 180°C/2 hrs, raise T to 204°C, hold 204°C/2 hrs |
| IME-3 | Same process as IME-1 | Same as IME-2 except flows at 85-120, gelation occurs at 134°C where pressure (0.69 MPa) was applied |
| IME-4 | Removed solvent at RT in vacuum over 72 hr period to produce resin powder | Same as IME-2 except flows at 40°C, gelation occurs at 160°C where pressure (0.69 MPa) was applied |
| IME-5 | Same process as IME-1 | 50°C in vacuum, raise T to 150°C, hold ½ hr, release vacuum, hold 150°C 1 hr, raise T to 180°C, hold 180°C/2 hrs, raise T to 204°C, hold 204°C/2 hrs |
| IME-6 | Same process as IME-4 | Same as IME-2 except no apparent flow, gelation occurs at 145°C, where pressure (0.69 MPa) was applied |
| IME-7 | Same process as IME-4 | Same as IME-2 except flows at 75°C, gelation at 148°C where pressure (0.69 MPa) was applied |
| IME-8 | Same process as IME-4 | Same as IME-2, except flows at 35°C, gelation occurs at 145°C, where pressure (0.69 MPa) was applied |

[1]All samples were postcured at 204°C for 24 hrs

Table 10

Some Physical Properties of Neat Resin Systems[1]

| Resin No. | Density[2] g/cc | Shrinkage[4] Due to Cure, % | Coefficient of Thermal Expansion[5] cm/cm/°C $\alpha \times 10^5$ |
|---|---|---|---|
| C | 1.27 | 0.81 | 5.15 |
| IME-1 | 1.36 | 0.81 | 5.00 |
| IME-2 | 1.28 | 0.90 | 6.45 |
| IME-3 | 1.30 | 0.99 | 7.02 |
| IME-4 | 1.33 | 0.90 | 6.10 |
| IME-5 | 1.33<br>1.34[3] | 0.84 | 5.15 |
| IME-6 | 1.26 | 0.70 | 5.17 |
| IME-7 | 1.29 | 0.80 | 6.45 |
| IME-8 | 1.29 | 0.90 | 6.26 |

[1] cure cycle: RT → gelation + 180°C/2 hrs + 204°C/2 hrs + postcure at 240°C/24 hrs

[2] from volume and weight measurement

[3] from liquid displacement technique

[4] after 2 hr cure at 204°C

[5] determined on a Theta Industries Dilatronic II Dilatometer

Table 11

Resin[1] Moisture Absorption

| Resin System | After RT 24 hrs in Water Wt % | Saturation at 180°C, 87% RH Wt % | After 72 hrs Water Boil Wt % |
|---|---|---|---|
| C | 0.34 | 3.4 | 4.03 |
| IME-1 | 0.41 | 2.4 | 3.72 |
| IME-2 | 0.41 | 1.6 | 2.31 |
| IME-3 | 0.41 | 1.4 | 2.43 |
| IME-4 | 0.36 | 1.8 | 3.13 |
| IME-5 | 0.44 | 1.8 | 3.70 |
| IME-6 | 0.74 | 1.8 | 2.47 |
| IME-7 | 0.90 | 2.0 | 2.66 |
| IME-8 | 0.59 | 2.5 | 3.44 |
| Narmco 5208 | 1.50 | 5.5 | - |
| DER 332/DDS | 1.00 | 4.1 | - |
| Shell X-801/DDS | - | 6.5 | - |

[1] all resin samples were postcured at 204°C for 24 hrs

Table 12

Resin[1] Thermal Properties

| Resin System | DSC Transitions, °C First | DSC Transitions, °C Second | TMA Transitions, °C First,Tg | TMA Transitions, °C Second | TGA Wt % Loss at (in air) 300°C | TGA Wt % Loss at (in air) 800°C |
|---|---|---|---|---|---|---|
| C | 125 | 255 | 184 | 227 | 1.0 | 70 |
| IME-1 | 95 | 260 | 196 | 215 | 1.5 | 61 |
| IME-2 | 100 | 250 | 185 | 215 | 1.7 | 60 |
| IME-3 | - | 250 | 192 | 220 | 0 | 56 |
| IME-4 | 245 | 290 | 207 | - | 2 | 55 |
| IME-5 | 100 | 255 | 192 | 220 | 2.5 | 60 |
| IME-6 | - | - | 193 | 217 | 1.4 | 58 |
| IME-7 | - | 245 | 190 | 212 | 2.0 | 66 |
| IME-8 | - | - | 211 | - | 3.0 | 58 |
| Typical Epoxy | - | - | 150 | 250 | 2.0 | 15 |

[1]all resins were postcured at 204°C for 24 hrs

Table 13

Resin[1] Char Yields

| Resin System | % Char Muffle[2] Furnace Method | TGA[3] Method |
|---|---|---|
| C | 20.7 | 20.0 |
| IME-1 | 39.3 | 39 |
| IME-2 | 33.8 | 40 |
| IME-3 | 39.9 | 44 |
| IME-4 | 29.4 | 45 |
| IME-5 | 34.2 | 40 |
| IME-6 | 35.8 | 42 |
| IME-7 | 31.3 | 34 |
| IME-8 | 30.8 | 42 |
| Hercules 3501-6 epoxy | 15.7 | - |

[1] all resins were postcured at 204°C for 24 hrs

[2] C&M high temperature muffle furnace for 3 min at 800°C

[3] DuPont 951 thermogravimetric analyzer in air at 800°C

Table 14

Resin[1] Tensile Properties

| Resin System | Tg, °C | Strength psi | Strength MPa | Modulus $10^6$ psi | Modulus GPa | Strain to Failure % |
|---|---|---|---|---|---|---|
| C MY720/DDS | 250 | 5966[2] | 41.1 | 0.60 | 4.13 | 0.99 |
| IME-1 MY720/6F-DDS | 240 | 7870 | 48.7 | 0.55 | 3.76 | 1.24 |
| IME-5 MY720/6F-DDS/DDS | 235 | 6500 | 44.8 | 0.70 | 4.82 | 0.99 |
| | | 6530 | 45.0 | 0.61 | 4.24 | 0.98 |
| Ciba-Geigy 6350 MY720/Dicyandiamide | 155 | 4643[2] | 29.9 | 0.63 | 4.34 | 0.80 |

[1]resins were postcured at 204°C for 24 hrs

[2]an average of five specimens

Table 15

Resin[1] Compression Strengths

| | Compression Strength | |
|---|---|---|
| | psi | MPa |
| C | 37,400 | 258 |
| IME-1 | 29,900 | 206 |
| -2 | 23,600 | 163 |
| -3 | 22,800 | 157 |
| -4 | 28,700 | 198 |
| -5 | 24,900 | 17.1 |
| -6 | 22,200 | 153 |
| -7 | 24,300 | 168 |
| -8 | 50,400 | 347 |
| Typical DDS cured epoxy | 34,000 | 234 |

[1]all resins were postcured at 204°C for 24 hrs

Table 16

Autoclave Process Parameters for Celion 6000
Epoxy Resin Composites

15.2 cm x 25.4 cm x 0.23 cm
10 plies

| Composite No. | Process Conditions |
|---|---|
| 1-C | Evacuation in vacuum bag at RT, then RT→140°C (2.0°C/min), at 140°C apply 0.69 MPa, hold 140°C/1 hr (0.69 MPa), 140°C→177°C (1.2°C/min), 0.69 MPa, hold 177°C/1 hr (0.69 MPa), 177°C→204°C (2.0°C/min), (0.69 MPa), hold 204°C/1 hr (0.69 MPa) |
| 2-IME-1 | Evacuation in vacuum bag at RT, then RT→122°C (1.6°C/min) at 122°C apply 0.69 MPa, hold 122°C/1 hr (0.69 MPa), 122°C→177°C (1.2°C/min), 0.69 MPa, hold 177°C/1 hr, 177°C→204°C (2.0°C/min), 0.69 MPa, hold 204°C/1 hr (0.69 MPa) |
| 3-IME-2 | Evacuation in vacuum bag at RT, then RT→122°C (1.6°C/min), at 122°C apply 1.38 MPa, hold 122°C/1 hr (1.38 MPa), 122°C→177°C (1.2°C/min), 1.38 MPa, hold 177°C/1 hr (1.38 MPa), 177°C→204°C (2.0°C/min), 1.38 MPa, hold 204°C/1 hr (1.38 MPa) |
| 4-IME-3 | Same cure cycle as 3-IME-2 |
| 5-IME-4 | Same cure cycle as 3-IME-2 and 4-IME-3 |
| 6-IME-5 | Evacuation in vacuum bag at RT, then RT→177°C (2.5°C/min), at 177°C apply 0.69 MPa, hold 177°C/1 hr (0.69 MPa), 177°C→204°C (2.0°C/min), 0.69 MPa, hold 204°C/1 hr (0.69 MPa) |
| 7-IME-6 | Same cure cycle as 3-IME-2, 4-IME-3 and 5-IME-4 |
| 8-IME-7 | Same cure cycle as 3-IME-2, 4-IME-3, 5-IME-4, and 7-IME-6 |
| 9-IME-8 | Same cure cycle as 3-IME-2, 4-IME-3, 5-IME-4, 7-IME-6 and 8-IME-7 |

Table 17

Celion 6000 Resin Composites
Compression Molded
(15.24 cm x 25.4 cm x 0.25 cm)
9 plies

| Composite No. | Processing Parameters |
|---|---|
| 33-C | RT→148°C, contact P (1.7°C/min) then at 148°C application of press.(1.38 MPa) to STOPS 148°C→177°C (0.8°C/min), hold, 177°C/2 hrs (pressure) + 204°C/2 hrs (air circulating oven) |
| 20-IME-1 | RT→150°C, contact P vacuum (1.4°C/min) at 150°C application of press.to STOPS, 150°C→177°C (1.7°C/min), hold 177°C/2 hrs (press.1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 34-IME-1 | RT→148°C, contact P (2.3°C/min) at 148°C application of pressure to STOPS, 148°C→177°C (0.5°C/min), hold, 177°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 21-IME-2 | RT→120°C (3.4°C/min) at 120°C application of press. (1.38 MPa) to STOPS, initially at RT, 120°C→150°C/1 hr (0.5°C/min), hold 1 hr, 150°C→177°C (2.1°C/min), hold 177°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 32-IME-2 | RT→136°C, contact P (2.0°C) at 136°C application of press. (1.38 MPa) to STOPS, 136°C→177°C (1.1°C/min), hold 177°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 36-IME-2 | RT→116°C (25 psi) (2.6°C/min), 116°C→128°C (2.4°C/min), contact P, then at 128°C application of press. (1.38 MPa) to STOPS, hold 5 min, 128°C→178°C (1.5°C/min), hold 178°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 22-IME-3 | RT→88°C contact P (2.3°C/min), then at 88°C application of press. (1.38 MPa) to STOPS, 88°C→177°C (1°C/min), hold 177°C/2 hrs (press.1.38 MPa), + 204°C/2 hrs (air circulating oven) |
| 28-IME-3 | RT→126°C, contact P (2°C/min), at 136°C application of press. (1.38 MPa) to STOPS, 126°C→177°C (1.4°C/min), hold 177°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |

Table 17 (Cont'd)

| Composite No. | Processing Parameters |
|---|---|
| 23-IME-4 | RT→150°C, contact P (2.3°C/min) at 150°C then application of press. (1.38 MPa) to STOPS, 150°C→177°C (1°C/min), hold 177°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 29-IME-4 | RT→145°C (1.9°C/min) 0.69 MPa initially, release P, 145°C→150°C, (1°C/min) (contact), at 150°C application of press. (1.38 MPa) to STOPS, 150°C→177°C (0.5°C/min), hold 177°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 24-IME-5 | RT→155°C, contact P (1.8°C/min), at 155°C application of press. (1.38 MPa) to STOPS, 155°C→177°C (1°C/min), hold 177°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 30-IME-5 | RT→130°C contact (1.8°C/min), at 130°C application of press. (1.38 MPa) to STOPS, 130°C→177°C (1.6°C/min), hold 177°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 25-IME-6 | RT→125°C contact (2.2°C/min), at 125°C application of press. to STOPS, 125°C→178°C (2°C/min), hold 178°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 31-IME-6 | RT→140°C contact P (2.0°C/min) at 140°C application of press. to STOPS, 140°C→178°C (1.5°C/min) (press, 1.38 MPa), hold 178°C/2 hrs, press. 1.38 MPa + 204°C/2 hrs (air circulating oven) |
| 26-IME-7 | RT→125°C contact P (2°C/min), at 125°C application of press. (1.38 MPa) to STOPS, 125°C→178°C (2.26°C/min), hold 178°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 35-IME-7 | RT→127°C contact P (2.1°C/min), at 127°C application of press. (1.38 MPa) to STOPS, hold 127°C, 5 min, 127°C→178°C (2.2°C/min) hold 178°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 37-IME-7 | RT→123°C contact P (2.2°C/min), at 123°C application of press. (1.38 MPa) to STOPS, hold 10 min, 123°C→178°C (2.0°C/min), hold 178°C/2 hrs (press, 1.38 MPa) + 204°C/2 hrs (air circulating oven) |

Table 17 (Cont'd)

| Composite No. | Processing Parameters |
|---|---|
| 27-IME-8 | RT→80°C contact P (1.7°C/min, 80°C→115°C (2.5°C/min), at 115°C application of pressure (1.38 MPa) to STOPS, 115°C→179°C (2.8°C/min), hold 177°C/2 hrs (press. 1.38 MPa) + 204°C/2 hrs (air circulating oven) |
| 38-IME-8 | RT→98°C, application of pressure 1.38 MPa (2.5°C/min), 98°C 106°C (2.0°C/min), pressure increased (2.76 MPa), 106°C→121°C (3.0°C/min), 4.85 MPa, 121°C→178°C (1.0°C/min), hold 178°C/2 hrs, pressured increased (4.85 MPa) + 204°C/2 hrs (air circulating oven) |

Table 18

Composition of Autoclave Molded Composites

(15.2 cm x 25.4 cm x 10 ply)

| Composite No. | Measured Density g/cc | Theoretical Density composite with 60 vol % Fiber g/cc | Theoretical Density composite with 70 vol % Fiber g/cc | Experimental[1] Vol % Resin | Experimental[1] Vol % Fiber | Experimental[1] Vol % Void | Composite Ply Thickness mm/ply |
|---|---|---|---|---|---|---|---|
| 1-C | 1.59 | 1.564 | 1.61 | 25.6 | 72.4 | 3.1 | 0.24 |
| | 1.59 | | | 26.9 | 70.7 | 2.5 | 0.24 |
| 2-IME-1 | 1.57 | 1.60 | 1.64 | 30.5 | 65.6 | 3.9 | 0.24 |
| | 1.57 | | | 29.7 | 66.2 | 4.0 | 0.24 |
| 3-IME-2 | 1.53 | 1.59 | 1.62 | 38.4 | 59.1 | 2.6 | 0.268 |
| | 1.54 | | | 34.9 | 61.9 | 3.2 | 0.264 |
| 4-IME-3 | 1.54 | 1.59 | 1.62 | 34.1 | 62.1 | 3.8 | 0.267 |
| | 1.55 | | | 32.2 | 63.9 | 3.9 | 0.263 |
| 5-IME-4 | 1.56 | 1.59 | 1.63 | 31.3 | 64.6 | 4.2 | 0.236 |
| | 1.55 | | | 34.8 | 61.4 | 3.9 | 0.24 |
| 6-IME-5 | 1.56 | 1.58 | 1.63 | 30.1 | 65.7 | 4.2 | 0.225 |
| | 1.55 | | | 26.8 | 68.0 | 5.3 | 0.216 |
| 7-IME-6 | 1.57 | 1.56 | 1.61 | 25.5 | 71.1 | 3.4 | 0.227 |
| | 1.54 | | | 28.4 | 67.3 | 4.3 | 0.221 |
| 8-IME-7 | 1.52 | 1.57 | 1.62 | 36.2 | 59.9 | 3.9 | 0.256 |
| | 1.50 | | | 33.6 | 60.8 | 5.6 | 0.246 |
| 9-IME-8 | 1.54 | 1.57 | 1.62 | 34.7 | 62.4 | 2.9 | 0.244 |
| | 1.55 | | | 33.3 | 63.4 | 3.3 | 0.235 |

[1] Acid digestion method

Table 19

Composition Data for Compression Molded Composites

(15.24 cm x 25.4 cm x 9 ply)

| Composite No. | Density g/cc | Calculated[1] Vol % Resin | Calculated[1] Vol % Fiber | Experimental[2] Vol % Resin | Experimental[2] Vol % Fiber | Experimental[2] Vol % Void | Composite Ply Thickness mm/ply |
|---|---|---|---|---|---|---|---|
| 33-C | 1.51 | 39.0 | 61.0 | 27.2 | 66.1 | 6.7 | 0.276 |
| | 1.54 | | | 36.1 | 61.4 | 2.6 | |
| 20-IME-1 | 1.58 | 42.9 | 57.1 | 38.2 | 60.4 | 1.3 | 0.289 |
| | 1.59 | | | 38.2 | 64.9 | 1.8 | |
| 34-IME-1 | 1.57 | 38.7 | 63.1 | 41.6 | 56.9 | 1.5 | 0.274 |
| | 1.56 | | | 40.2 | 57.4 | 2.4 | |
| 21-IME-2 | 1.58 | 40.7 | 59.3 | 33.4 | 65.5 | 1.1 | 0.297 |
| | 1.57 | | | 41.1 | 53.4 | - | |
| 32-IME-2 | 1.52 | 39.2 | 60.8 | 40.3 | 57.2 | 2.6 | 0.276 |
| | 1.54 | | | 40.0 | 58.2 | 1.8 | |
| 36-IME-2 | 1.54 | 41.6 | 58.4 | 37.8 | 60.3 | 1.9 | 0.279 |
| | 1.56 | | | 34.8 | 63.3 | 1.9 | |
| 28-IME-3 | 1.54 | 40.5 | 59.5 | 42.8 | 55.5 | 1.8 | 0.282 |
| | 1.54 | | | 31.5 | 63.9 | 4.6 | |
| 27-IME-4 | 1.55 | 40.2 | 59.8 | 39.6 | 58.1 | 2.4 | 0.287 |
| | 1.57 | | | 42.8 | 56.4 | 0.80 | |
| 29-IME-4 | 1.56 | 40.5 | 59.5 | 42.1 | 56.6 | 1.3 | 0.279 |
| | 1.56 | | | 41.5 | 57.3 | 1.2 | |
| 24-IME-5 | 1.55 | 37.4 | 62.6 | 34.9 | 62.1 | 2.9 | 0.272 |
| | 1.56 | | | - | - | - | |
| 30-IME-5 | 1.55 | 37.2 | 62.8 | 33.8 | 62.4 | 3.8 | 0.269 |
| | 1.55 | | | 37.6 | 59.7 | 2.7 | |
| 25-IME-6 | 1.52 | 38.7 | 61.3 | 36.9 | 59.3 | 3.8 | 0.282 |
| | 1.50 | | | - | - | - | |

Table 19 (Cont'd)

| Composite No. | Density g/cc | Calculated[1] Vol % Resin | Calculated[1] Vol % Fiber | Experimental[2] Vol % Resin | Experimental[2] Vol % Fiber | Experimental[2] Vol % Void | Composite Ply Thickness mm/ply |
|---|---|---|---|---|---|---|---|
| 31-IME-6 | 1.55 | 39.6 | 60.4 | 42.8 | 57.1 | 0.04 | 0.276 |
| | 1.54 | | | 42.3 | 57.1 | 0.54 | |
| 26-IME-7 | 1.49 | 38.4 | 61.6 | 38.8 | 56.4 | 4.9 | 0.276 |
| | 1.52 | | | 30.4 | 64.0 | 5.6 | |
| 35-IME-7 | 1.55 | 41.4 | 58.6 | 35.6 | 61.7 | 2.7 | 0.297 |
| | 1.54 | | | 37.8 | 59.8 | 2.4 | |
| 37-IME-7 | 1.55 | 41.8 | 58.2 | 37.7 | 60.2 | 2.1 | 0.274 |
| | 1.54 | | | 38.9 | 59.1 | 1.9 | |
| 27-IME-8 | 1.52 | 40.5 | 59.5 | 39.1 | 57.8 | 3.1 | 0.284 |
| | 1.52 | | | 40.4 | 56.9 | 2.6 | |
| 38-IME-8 | 1.55 | 40.9 | 59.1 | 37.2 | 60.9 | 1.9 | 0.274 |
| | 1.58 | | | 38.0 | 61.8 | 0.20 | |

[1]based on data: Finished composite weight = initial weight of 9 plies (fiber + resin) - weight of resin flash removed. Fiber weight of each composite = 105.246g, density of fiber = 1.76g. Resin density varies with each resin. Calculation assumes zero void content.

[2]Acid digestion method

Table 20

Moisture Absorption of Celion 6000 Epoxy Resin Composites

| | After 24 hrs RT Wt % | Saturation at 82°C, 87% RH Wt % | | After 72 hrs Water Boil Wt % |
|---|---|---|---|---|
| | flex. spec. | shear spec. | flex. spec. | flex. spec. |
| 1-C | 0.05 | 0.65 | 0.45 | 0.89 |
| 2-IME-1 | 0.09 | 0.54 | 0.51 | 1.05 |
| 3-IME-2 | 0.08 | 0.35 | 0.36 | 0.62 |
| 4-IME-3 | 0.12 | 0.42 | 0.36 | 1.21 |
| 5-IME-4 | 0.15 | 0.61 | 0.53 | 0.86 |
| 6-IME-5 | 0.09 | 0.63 | 0.59 | 0.85 |
| 7-IME-6 | 0.16 | 0.62 | 0.56 | 0.88 |
| 8-IME-7 | 0.09 | 0.69 | 0.60 | 0.69 |
| 9-IME-8 | 0.11 | 0.76 | 0.62 | 1.15 |
| 33-C | 0.25 | 1.09 | 0.69 | 1.20 |
| 20-IME-1 | 0.12 | 0.66 | 0.56 | 0.89 |
| 34-IME-1 | 0.07 | - | - | 0.99 |
| 21-IME-2 | 0.13 | - | - | 0.65 |
| 32-IME-2 | 0.12 | 0.50 | 0.32 | 0.74 |
| 36-IME-2 | 0.10 | - | - | 0.70 |
| 28-IME-3 | 0.21 | 0.57 | 0.41 | 0.71 |
| 23-IME-4 | 0.17 | - | - | 1.17 |
| 29-IME-4 | 0.15 | 0.67 | 0.57 | 0.91 |
| 24-IME-5 | 0.09 | 0.65 | 0.64 | 1.10 |
| 30-IME-5 | 0.12 | 0.72 | 0.63 | 1.06 |
| 25-IME-6 | 0.18 | - | - | 0.89 |
| 31-IME-6 | 0.14 | 0.58 | 0.42 | 0.60 |
| 26-IME-7 | 0.24 | 0.88 | 0.39 | 1.10 |
| 35-IME-7 | 0.09 | - | - | 0.82 |
| 37-IME-7 | 0.10 | - | - | 0.76 |
| 11-C | 0.13 | 0.58 | 0.58 | 1.16 |
| 13-IME-2 | 0.09 | 0.39 | 0.46 | 0.69 |
| 14-IME-3 | 0.15 | 0.36 | 0.43 | 0.87 |
| 15-IME-4 | 0.14 | 0.48 | 0.60 | 1.05 |
| 17-IME-6 | 0.14 | 0.20 | 0.41 | 1.15 |
| 18-IME-7 | 0.07 | 0.44 | 0.52 | 0.60 |

Table 21

Thermal Characteristics of Celion 6000 Epoxy Resin Composites[1]

| | TMA Transitions, °C | | TGA Wt % Loss at (in air) | |
|---|---|---|---|---|
| | First, Tg | Second | 300°C | 800°C |
| 1-C | 230 | 275 | 0.6 | 15 |
| 33-C | - | 275 | 0.2 | 17 |
| 2-IME-1 | - | 285 | 0.7 | 14 |
| 34-IME-1 | 175 | 285 | 0.8 | 21 |
| 3-IME-2 | - | 180 | 0.5 | 17 |
| 32-IME-2 | 175 | 275 | 0.7 | 17 |
| 4-IME-3 | - | 210 | 0.4 | 15 |
| 28-IME-3 | 195 | 235 | 1.0 | 20 |
| 5-IME-4 | 145 | 265 | 0.6 | 15 |
| 23-IME-4 | 150 | 235 | 0.2 | 18 |
| 29-IME-4 | - | 240 | 0.5 | 19 |
| 6-IME-5 | - | - | 0.5 | 14 |
| 24-IME-5 | - | 225 | 0.4 | 19 |
| 30-IME-5 | - | 255 | 1.0 | 18 |
| 7-IME-6 | 160 | 275 | 0.5 | 13 |
| 31-IME-6 | 185 | 225 | 0.4 | 17 |
| 8-IME-7 | 205 | 250 | 0.5 | 17 |
| 26-IME-7 | 180 | 250 | 1.0 | 18 |
| 35-IME-7 | 180 | 250 | 0.8 | 27 |
| 37-IME-7 | 180 | 265 | 0.7 | 26 |
| 9-IME-7 | 175 | 280 | 0.8 | 17 |
| 27-IME-8 | 145 | 245 | 0.4 | 21 |
| 38-IME-8 | 150 | 245 | 2.0 | 16 |

[1] composites were postcured at 204°C for 24 hrs

Table 22

Char Forming Properties of Celion 6000/Epoxy Resin Composites

| Composite System | Wt % Retention Due to Pyrolysis Wt % TGA Method[1] | Muffle Furnace Method[2] | Wt % Resin Char Yield in Composite[3] | Wt % Fiber Containment[4] |
|---|---|---|---|---|
| C | 85 | 84 | 15 | 99 |
| -1 | 86 | 87 | 46 | 101 |
| -2 | 83 | 75 | 41 | 111 |
| -3 | 85 | 83 | 47 | 104 |
| -4 | 84 | 80 | 35 | 100 |
| -5 | 86 | 84 | 33 | 95 |
| -6 | 87 | 75 | 41 | 103 |
| -7 | 83 | 81 | 44 | 106 |
| -8 | 83 | 78 | 30 | 94 |

[1] DuPont 951 thermogravimetric analyzer in air at 800°C

[2] C&M high temperature muffle furnaces for 3 min at 800°C

[3] $\%\ \text{resin char yield} = \dfrac{\text{wt char after burning}}{\text{wt resin before burning}} \times 100$

Wt % resin before and after burning were determined by $H_2O_4/H_2O_2$ digesting of composites before and after burning

[4] $\%\ \text{fiber containment} = \dfrac{\text{wt fiber after burning}}{\text{wt fiber before burning}} \times 100$

Wt % fiber before and after burning were determined by $H_2SO_4/H_2O_2$ digesting of composites before and after burning

Table 23

Interlaminar Shear Strengths[1] of Celion 6000/Resin Composites[2]
(Dry Condition)

| Composite No.[2] | RT Interlaminar Shear Strength[1] "as-fabricated"[3] psi | MPa | 3 Hr Postcure @ 477 K (204°C) psi | MPa | 24 Hr Postcure @ 477 K (204°C) psi | MPa |
|---|---|---|---|---|---|---|
| 1-C | 15,000[4] | 107 | 15,000 | 104 | 15,300 | 106.0 |
| 2-IME-1 | 10,400[4] | 71.6 | 10,800 | 74.5 | 8,200 | 56.5 |
| 3-IME-2 | 13,840[5] | 95.6 | 12,600 | 86.9 | 12,700 | 87.3 |
| 4-IME-3 | 13,360[5] | 92.0 | 12,500 | 86.2 | 12,500 | 85.8 |
| 5-IME-4 | 12,000[4] | 82.6 | 11,700 | 80.5 | 11,600 | 79.8 |
| 6-IME-5 | 15,000[5] | 103.2 | 16,300 | 112.0 | 14,100 | 97.4 |
| 7-IME-6 | 9,610[4] | 66.3 | 8,230 | 56.8 | 9,790 | 67.5 |
| 8-IME-7 | 13,340[5] | 91.9 | 13,000 | 89.4 | 13,300 | 91.8 |
| 9-IME-8 | 11,880[5] | 81.8 | 11,600 | 80.2 | 12,400 | 85.5 |

[1] span-to-depth ratio, S/D = 4/1

[2] composites were autoclave molded, for cure cycle see Table 16

[3] composites were not postcured

[4] 1 specimen tested

[5] an average of 5 tests

Table 24

Interlaminar Shear Strengths[1] of Celion 6000/
Epoxy Resin Composites[2,3]

(Dry Condition)

| | Interlaminar Shear Strength | | | | | |
|---|---|---|---|---|---|---|
| | RT | | 423 K (150°C)[4] | | 450 K (177°C)[4] | |
| | psi | MPa | psi | MPa | psi | MPa |
| 1-C | 13,600 | 93.8 | 10,100 | 69.6 | 9,290 | 64.1 |
| | 13,300 | 92.0 | 9,690 | 66.8 | 7,940 | 54.7 |
| 2-IME-1 | 8,480 | 58.4 | 5,950 | 41.0 | 5,570 | 38.4 |
| | 8,980 | 61.4 | 5,750 | 39.6 | 5,740 | 39.6 |
| 3-IME-2 | 11,400 | 78.4 | 6,860 | 47.3 | 5,020 | 34.6 |
| | 11,600 | 79.6 | 6,970 | 48.0 | 5,170 | 35.6 |
| 4-IME-3 | 11,500 | 79.0 | 6,400 | 44.2 | 5,030 | 34.6 |
| | 11,300 | 77.7 | 7,030 | 48.5 | 4,800 | 33.1 |
| 5-IME-4 | 10,000 | 69.3 | 6,590 | 45.5 | 5,360 | 36.9 |
| | 10,400 | 71.6 | 6,400 | 44.1 | 5,320 | 36.7 |
| 6-IME-5 | 12,900 | 89.2 | 6,960 | 48.0 | 8,200 | 56.6 |
| | 12,700 | 87.4 | 7,360 | 50.7 | 8,222 | 56.7 |
| 7-IME-6 | 8,240 | 56.8 | 5,060 | 34.9 | 4,740 | 32.6 |
| | 8,090 | 55.8 | 5,370 | 37.1 | 3,990 | 27.5 |
| 8-IME-7 | 11,600 | 80.2 | 7,850 | 54.1 | 6,710 | 46.3 |
| | 11,700 | 80.9 | 7,210 | 49.7 | 6,600 | 45.5 |
| 9-IME-8 | 10,500 | 72.7 | 6,270 | 43.2 | 4,840 | 33.4 |
| | 9,820 | 67.7 | 6,370 | 43.9 | 5,820 | 40.1 |

[1] span-to-depth ratio 4/1

[2] composites were autoclave cured, for cure cycle see Table 16

[3] all composites postcured @ 477 K (204°C) for 24 hrs

[4] after 20 min soak at temperature

Table 25

Interlaminar Shear Strength[1] of Humidity Exposed[2] Celion 6000/Epoxy Resin Composites[3,4]

| | Interlaminar Shear Strength | | | | | |
|---|---|---|---|---|---|---|
| | RT | | 423 K (150°C)[5] | | 450 K (177°C)[5] | |
| | psi | MPa | psi | MPa | psi | MPa |
| 1-C | 11,000 | 75.9 | 6,125 | 42.2 | 5,310 | 36.6 |
| | 11,900 | 81.9 | 6,090 | 42.0 | 4,680 | 32.3 |
| 2-IME-1 | 7,220 | 49.8 | 5,300 | 36.6 | 5,210 | 35.9 |
| | 7,490 | 51.6 | 5,220 | 36.0 | 5,060 | 34.9 |
| 3-IME-2 | 10,500 | 72.2 | 4,700 | 32.4 | 3,500 | 24.2 |
| | 10,500 | 72.4 | 4,460 | 30.7 | 3,070 | 21.2 |
| 4-IME-3 | 10,300 | 70.8 | 4,660 | 32.1 | 3,140 | 21.7 |
| | 10,100 | 69.8 | 4,770 | 32.9 | 3,380 | 23.3 |
| 5-IME-4 | 10,300 | 70.7 | 4,390 | 30.3 | 3,040 | 21.0 |
| | 10,200 | 70.2 | 4,750 | 32.8 | 3,120 | 21.5 |
| 6-IME-5 | 12,100 | 83.4 | 5,800 | 40.0 | 4,080 | 28.1 |
| | 10,100 | 69.3 | 5,350 | 36.9 | 4,410 | 30.4 |
| 7-IME-6 | 7,410 | 51.1 | 3,990 | 27.5 | 3,870 | 26.7 |
| | 7,740 | 53.4 | 4,720 | 32.6 | 3,470 | 23.9 |
| 8-IME-7 | 9,260 | 63.8 | 5,260 | 36.3 | 3,520 | 24.3 |
| | 9,570 | 66.0 | 5,130 | 35.3 | 3,900 | 26.9 |
| 9-IME-8 | 9,420 | 65.0 | 3,930 | 27.1 | 2,620 | 18.1 |
| | 9,580 | 66.1 | 3,530 | 24.4 | 2,830 | 19.5 |

[1] span-to-depth 4/1

[2] 87% RH at 355 K (82°C) to saturation

[3] all composites autoclave cured, for cure cycle see Table 16

[4] composites were postcured at 477 K (204°C) for 24 hrs

[5] after 20 min soak at temperature

Table 26

Interlaminar Shear Strengths[1] of Celion 6000/
IME Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | Interlaminar Shear Strength | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | "as-fabricated" | | | | | | 3 hr postcure @ 477 K (204°C) | |
| | RT | | 450 K (177°C)[4] | | 477 K (204°C)[4] | | 423 K (150°C)[4] | |
| | psi | MPa | psi | MPa | psi | MPa | psi | MPa |
| 11-IME-1 | 16,200 | 111 | 8,540 | 58.9 | 4,650 | 32.1 | 9,440 | 67.1 |
| | 15,600 | 108 | 7,660 | 52.8 | | | | |
| 13-IME-2 | 12,900 | 88.9 | 4,260 | 29.4 | 2,220 | 15.2 | 6,080 | 42.0 |
| | 12,700 | 87.7 | 4,510 | 31.1 | | | | |
| 14-IME-3 | 11,900 | 82.1 | 4,210 | 29.0 | - | - | 6,560 | 45.2 |
| 15-IME-4 | 12,800 | 88.3 | 5,360 | 36.9 | - | - | 7,370 | 50.8 |
| 17-IME-6 | 9,890 | 68.2 | 3,420 | 23.6 | - | - | 5,330 | 36.7 |
| 18-IME-7 | 11,900 | 82.1 | 3,930 | 27.1 | - | - | 5,220 | 36.0 |

[1]Short beam shear strength, S/D = 4/1

[2]Composites (10cm x 10cm x ~.254cm) were compression molded, similar to the process described in Table 17 for larger composites

[3]Composites were not postcured

[4]after 20 min soak at temperature

Table 27

Interlaminar Shear Strengths[1] of Celion 6000/Epoxy Resin Composites[2]
(Dry Condition)

| Composite No. | Interlaminar Shear Strength | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | as fabricated[3] | | | | | | 3 hr postcure @ 477 K (204°C) | |
| | RT | | 450 K (177°C)[4] | | 477 K (204°C)[4] | | 423 K (150°C)[4] | |
| | RT | MPa | psi | MPa | psi | MPa | psi | MPa |
| 33-C | 17,300 | 119 | 10,400 | 71.7 | 5080 | 35.0 | 9100 | 62.8 |
| | 15,400 | 106 | 6,700 | 46.2 | 5680 | 39.2 | | |
| | 16,100 | 111 | 7,210 | 49.7 | 4820 | 33.2 | | |
| 20-IME-1 | 16,900 | 117 | 7,420 | 51.1 | 4650 | 32.0 | 9330 | 64.1 |
| | 15,000 | 104 | 7,720 | 53.2 | 6270 | 43.2 | | |
| | 14,200 | 97.9 | | | | | | |
| 34-IME-1 | 14,000 | 96.4 | - | - | - | - | 7630 | 52.6 |
| 32-IME-2 | 13,500 | 93.1 | 2,330 | 16.1 | 1570 | 10.8 | 4670 | 32.2 |
| | 12,000 | 82.7 | 2,500 | 17.8 | 1770 | 12.2 | | |
| | 11,700 | 80.6 | | | | | | |
| 28-IME-3 | 12,000 | 82.6 | 2,810 | 19.4 | 1520 | 10.5 | 4870 | 33.5 |
| | 11,900 | 82.1 | 2,940 | 20.3 | 1690 | 11.7 | | |
| 23-IME-4 | - | - | - | - | - | - | 8080 | 55.7 |
| 29-IME-4 | 11,500 | 79.3 | 6,380 | 44.0 | 3830 | 26.4 | 7430 | 51.2 |
| | 12,000 | 86.5 | 5,810 | 40.0 | 4050 | 27.9 | | |
| | 11,900 | 82.1 | 5,740 | 39.6 | 3340 | 23.0 | - | - |
| 24-IME-5 | 17,200 | 119 | 10,600 | 72.7 | 5290 | 36.4 | 9050 | 62.4 |
| | 15,300 | 106 | 6,330 | 43.6 | 4700 | 32.4 | | |
| | 15,100 | 104 | | | | | | |
| | 15,400 | 106 | 5,920 | 40.8 | 4050 | 27.9 | - | - |
| 30-IME-5 | - | - | - | - | - | - | 5670 | 39.1 |
| 25-IME-6 | 12,000 | 82.9 | 3,610 | 24.9 | 2050 | 14.1 | 5060 | 34.9 |
| | 10,800 | 74.3 | 3,730 | 25.7 | 2270 | 15.7 | | |
| 31-IME-6 | 11,100 | 76.6 | - | - | - | - | 5670 | 39.1 |
| 26-IME-7 | 12,900 | 88.6 | 4,440 | 30.6 | 2900 | 20.0 | 5890 | 40.6 |
| | 12,700 | 87.6 | 4,950 | 34.1 | 3160 | 21.8 | | |
| 27-IME-8 | 12,900 | 89.2 | 8,140 | 52.4 | 2180 | 15.0 | 4940 | 34.1 |
| | 11,500 | 79.3 | 3,390 | 23.3 | 2210 | 15.2 | | |
| | 12,200 | 84.0 | | | | | | |

[1] span-to-depth ratio, S/D = 4/1
[2] compression molded composites, for cure cycle see Table 17
[3] composites were not postcured
[4] after 20 min soak at temperature

Table 28

Interlaminar Shear Strength[1] of Celion 6000/
Epoxy Resin Composites[2,3]
(After 72 hr Boiling Water Exposure)

| Composite No. | Interlaminar Shear Strength | | | | | |
|---|---|---|---|---|---|---|
| | RT | | 450 K (177°C)[4] | | 477 K (204°C)[4] | |
| | ksi | MPa | ksi | MPa | ksi | MPa |
| 33-C | 10,500 | 72.5 | 3540 | 24.4 | 2480 | 17.1 |
| | 11,700 | 80.5 | 3780 | 26.0 | 1730 | 11.9 |
| 20-IME-1 | 10,300 | 70.8 | 3980 | 27.4 | 4020 | 24.7 |
| | 12,500 | 86.4 | 4740 | 92.7 | 3580 | 16.9 |
| 32-IME-2 | 9,900 | 68.2 | 2070 | 14.4 | 1400 | 9660 |
| | 11,300 | 78.1 | 2111 | 14.6 | 1310 | 9020 |
| 28-IME-3 | 8,940 | 61.6 | 2410 | 16.6 | 1800 | 12.4 |
| | 9,690 | 66.8 | 2340 | 16.1 | 2630 | 18.1 |
| 29-IME-4 | 9,920 | 68.4 | 4120 | 28.4 | 1790 | 12.3 |
| | 10,300 | 71.2 | 2260 | 15.6 | 1610 | 11.0 |
| 24-IME-5 | 10,400 | 71.7 | 6910 | 47.6 | 2450 | 16.9 |
| | 9,980 | 68.8 | 2850 | 19.7 | 3070 | 21.2 |
| 25-IME-6 | 8,260 | 56.9 | 2930 | 20.6 | 2100 | 14.5 |
| | 8,760 | 60.4 | 2880 | 19.9 | 1950 | 13.4 |
| 26-IME-7 | 8,940 | 61.7 | 2920 | 20.1 | 2740 | 18.9 |
| | 8,340 | 57.5 | 2790 | 19.2 | 2060 | 14.2 |
| 27-IME-8 | 8,830 | 60.9 | 2470 | 17.0 | 1730 | 11.9 |
| | 7,400 | 51.0 | 2550 | 17.6 | 1900 | 13.2 |

[1] span-to-depth ratio, 4/1

[2] composites were compression molded, for cure cycle see Table 17

[3] composites were not postcured

[4] after 20 min soak at temperature

Table 29

Interlaminar Shear Strengths[1] of
Celion 6000/IME Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | Interlaminar Shear Strength | | | | | |
|---|---|---|---|---|---|---|
| | RT | | 423 K (150°C)[4] | | 450 K (177°C)[4] | |
| | ksi | MPa | ksi | MPa | ksi | MPa |
| 33-C | 13,700 | 94.5 | 9,720 | 67.0 | 8530 | 58.8 |
| | | | 10,000 | 69.1 | 8290 | 57.2 |
| 20-IME-1 | 14,800 | 102 | 10,600 | 72.9 | 9250 | 63.8 |
| | | | 10,500 | 72.6 | 9560 | 65.9 |
| 34-IME-1 | 13,300 | 92.0 | 8,830 | 60.9 | 7888 | 54.3 |
| 32-IME-2 | 12,300 | 84.7 | 5,680 | 39.2 | 4640 | 32.0 |
| | | | 5,750 | 39.6 | 4410 | 30.4 |
| 36-IME-2 | 10,900 | 75.0 | 5,970 | 41.1 | 4670 | 32.2 |
| 28-IME-3 | 12,200 | 84.4 | 6,840 | 47.1 | 6120 | 42.2 |
| | | | 6,760 | 46.6 | 5460 | 37.6 |
| 29-IME-4 | 10,500 | 72.0 | 8,810 | 60.7 | 7720 | 53.2 |
| | | | 7,830 | 54.0 | 7860 | 54.2 |
| 24-IME-5 | 16,200 | 112 | 10,300 | 70.8 | 7790 | 53.7 |
| | 16,100 | 111 | 10,600 | 71.4 | 7480 | 51.6 |
| 30-IME-5 | 16,600 | 114 | 10,600 | 74.4 | 8060 | 55.6 |
| | | | 10,800 | 74.5 | 7690 | 53.0 |
| 31-IME-6 | 12,200 | 84.4 | 6,760 | 46.6 | 5230 | 36.0 |
| | | | 6,830 | 47.1 | 5300 | 36.0 |
| 26-IME-7 | 11,200 | 76.9 | 6,800 | 46.9 | 6190 | 42.6 |
| | - | - | - | - | 5710 | 39.4 |
| 35-IME-7 | 10,900 | 75.3 | 7,030 | 48.5 | 5700 | 39.3 |
| 37-IME-7 | 11,200 | 77.2 | 6,520 | 45.0 | 5300 | 36.6 |
| 27-IME-8 | 13,300 | 92.0 | 5,860 | 40.4 | 5570 | 38.4 |
| | 12,600 | 87.2 | 6,740 | 46.4 | 4900 | 33.8 |
| 38-IME-8 | 4,210 | 29.0 | - | - | - | - |
| | 4,020 | 27.7 | - | - | - | - |

[1] span-to-depth ratio 4/1
[2] composites were compression molded for cure cycle, see Table 17
[3] all composites postcured at 477 K (204°C) for 24 hrs

[4] after 20 min soak at temperature

Table 30

Interlaminar Shear Strength[1] of Humidity Exposed[2] Celion 6000/IME-Epoxy Resin Composites[3,4]

| Composite No. | Interlaminar Shear Strength | | | | | |
|---|---|---|---|---|---|---|
| | RT | | 423 K (150°C)[5] | | 450 K (177°C)[5] | |
| | ksi | MPa | ksi | MPa | ksi | MPa |
| 33-C | 8,360 | 57.6 | 5270 | 36.3 | 4130 | 28.5 |
| | 11,100 | 76.4 | 5320 | 36.7 | 4050 | 27.9 |
| 20-IME-1 | 10,200 | 70.5 | 6930 | 47.8 | 4900 | 33.8 |
| | 10,700 | 73.6 | 4900 | 33.8 | 5100 | 35.8 |
| 32-IME-2 | 9,500 | 65.5 | 3930 | 27.1 | 2460 | 16.9 |
| | 9,760 | 67.3 | 4500 | 31.0 | 2570 | 17.7 |
| 28-IME-3 | 10,100 | 69.4 | 4090 | 28.2 | 3430 | 23.6 |
| | 10,200 | 70.4 | 4160 | 28.7 | 3202 | 22.1 |
| 29-IME-4 | 9,640 | 66.4 | 4850 | 33.5 | 3860 | 26.6 |
| | 9,380 | 64.6 | 4670 | 32.2 | 3960 | 27.3 |
| 24-IME-5 | 13,600 | 93.9 | 7320 | 50.5 | 4520 | 31.2 |
| | 13,600 | 93.9 | 7220 | 49.8 | 4460 | 30.7 |
| 30-IME-5 | 13,500 | 93.0 | 7120 | 49.1 | 4930 | 34.0 |
| | | | 7300 | 50.3 | 5060 | 34.9 |
| 31-IME-6 | 9,600 | 66.2 | 4270 | 29.5 | 3210 | 22.2 |
| | 9,530 | 65.7 | 4520 | 31.2 | 3150 | 21.7 |
| 26-IME-7 | 9,940 | 68.5 | 4820 | 33.2 | 3940 | 27.2 |
| | 9,280 | 64.0 | 4990 | 34.4 | 3660 | 25.2 |
| 27-IME-8 | 11,400 | 78.8 | 5090 | 35.1 | 3130 | 21.6 |
| | 11,400 | 78.8 | 5220 | 36.0 | 3140 | 21.6 |

[1] span-to-depth ratio, 4/1

[2] 87% RH at 355 K (82°C) to saturation

[3] composites were compression molded, for cure cycle see Table 17

[4] composites were postcured at 477 K (204°C) for 24 hrs

[5] after 20 min soak at temperature

Table 31

RT Flexural Properties[1] of Celion 6000/Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | As-Fabricated[4] Strength ksi | As-Fabricated[4] Strength MPa | As-Fabricated[4] Modulus $10^6$psi | As-Fabricated[4] Modulus GPa | Postcured 3 hrs @ 477 K (204°C) Strength ksi | Postcured 3 hrs @ 477 K (204°C) Strength MPa | Postcured 3 hrs @ 477 K (204°C) Modulus $10^6$psi | Postcured 3 hrs @ 477 K (204°C) Modulus GPa | Postcured 24 hrs @ 477 K (204°C) Strength ksi | Postcured 24 hrs @ 477 K (204°C) Strength MPa | Postcured 24 hrs @ 477 K (204°C) Modulus $10^6$psi | Postcured 24 hrs @ 477 K (204°C) Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1-C | 235 | 1622 | 16.8 | 116 | 232 | 1603 | 18.1 | 125 | 259 | 1787 | 19.7 | 136 |
| 2-IME-1 | 222 | 1528 | 19.3 | 133 | 211 | 1453 | 17.6 | 122 | 195 | 1344 | 18.8 | 129 |
| 3-IME-2 | 187[5] | 1292 | 16.8 | 116 | 183 | 1265 | 18.2 | 125 | 172 | 1184 | 16.0 | 110 |
| 4-IME-3 | 207[5] | 1425 | 17.4 | 120 | 238 | 1640 | 16.2 | 112 | 185 | 1274 | 16.1 | 111 |
| 5-IME-4 | 213 | 1467 | 17.5 | 121 | 201 | 1385 | 17.1 | 118 | 197 | 1359 | 16.3 | 112 |
| 6-IME-5 | 218 | 1502 | 16.4 | 113 | 246 | 1696 | 17.3 | 120 | 219 | 1513 | 17.5 | 121 |
| 7-IME-6 | 229 | 1581 | 19.0 | 131 | 233 | 1606 | 19.4 | 134 | 203 | 1400 | 15.8 | 109 |
| 8-IME-7 | 192[5] | 1323 | 16.4 | 113 | 178 | 1225 | 16.0 | 110 | 219 | 1508 | 16.9 | 117 |
| | 52.9[6] | 364 | - | - | 118[6] | 814 | 15.5 | 107 | | | | |
| | 45.3[7] | 312 | - | - | | | | | | | | |
| 9-IME-8 | 203[5] | 1399 | 18.1 | 125 | 198 | 1365 | 17.2 | 119 | 200 | 1376 | 16.7 | 115 |

[1] Three point flex test at a span-to-depth ratio, 20/1
[2] Composites were autoclave molded, for cure cycle see Table 16
[3] Single specimen tested unless otherwise noted
[4] Composites were not postcured
[5] Average of 5 specimens
[6] 450 K (177°C) value
[7] 477 K (204°C) value

Table 32

Flexural Properties[1] of Postcured Celion 6000/Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | RT Strength[4] ksi | RT Strength[4] MPa | RT Modulus $10^6$psi | RT Modulus GPa | 423 K (150°C)[5] Strength[4] ksi | 423 K (150°C)[5] Strength[4] MPa | 423 K (150°C)[5] Modulus $10^6$psi | 423 K (150°C)[5] Modulus GPa | 450 K (177°C)[5] Strength[4] ksi | 450 K (177°C)[5] Strength[4] MPa | 450 K (177°C)[5] Modulus $10^6$psi | 450 K (177°C)[5] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1-C | 212 | 1460 | 16.3 | 112 | 230 | 1583 | 20.7 | 97.9 | 206 | 1421 | 18.9 | 131 |
| | 265 | 1826 | 16.5 | 114 | 175 | 1205 | 18.2 | 143 | 192 | 1327 | 19.1 | 132 |
| 2-IME-1 | 233 | 1608 | 18.1 | 125 | 117 | 805 | 17.6 | 121 | 167 | 1150 | 18.2 | 126 |
| | 122 | 841 | 17.3 | 119 | 159 | 1094 | 19.3 | 133 | 139 | 961 | 17.0 | 117 |
| 3-IME-2 | 165 | 1139 | 16.5 | 114 | 122 | 841 | 16.4 | 113 | 106 | 729 | 15.0 | 104 |
| | 167 | 1154 | 17.2 | 118 | 104 | 714 | 14.6 | 100 | 87.0 | 600 | 14.6 | 100 |
| 4-IME-3 | 164 | 1131 | 17.2 | 119 | 114 | 785 | 14.2 | 97.7 | 48.2 | 332 | 11.0 | 75.8 |
| | 177 | 1220 | 16.9 | 116 | 111 | 764 | 14.8 | 102 | 67.7 | 466 | 12.1 | 83.4 |
| 5-IME-4 | 170 | 1175 | 16.8 | 115 | 126 | 871 | 14.9 | 103 | 71.8 | 495 | 11.9 | 82.1 |
| | 169 | 1168 | 14.7 | 101 | 130 | 899 | 15.5 | 107 | 69.1 | 476 | 6.06 | 41.8 |
| 6-IME-5 | 199 | 1372 | 18.4 | 127 | 180 | 1238 | 17.5 | 121 | 123 | 848 | 7.67 | 52.8 |
| | 223 | 1536 | 20.1 | 139 | 193 | 1329 | 18.4 | 127 | 117 | 809 | 7.51 | 51.8 |
| 7-IME-6 | 178 | 1229 | 18.9 | 130 | 146 | 1005 | 18.7 | 129 | 61.3 | 423 | 7.15 | 49.3 |
| | 191 | 1316 | 18.5 | 128 | 144 | 995 | 18.5 | 128 | 70.6 | 487 | 7.43 | 51.2 |
| 8-IME-7 | 195 | 1346 | 17.7 | 122 | 135 | 934 | 15.8 | 109 | 76.3 | 526 | 6.99 | 48.2 |
| | 183 | 1264 | 17.3 | 119 | 120 | 827 | 15.1 | 104 | 86.2 | 594 | 6.94 | 47.9 |
| 9-IME-8 | 183 | 1260 | 17.1 | 118 | 132 | 908 | 15.7 | 108 | 65.6 | 452 | 6.41 | 44.2 |
| | 204 | 1408 | 17.2 | 119 | 123 | 846 | 16.0 | 110 | 73.9 | 509 | 6.23 | 43.0 |

[1] Three point flex test at a span-to-depth ratio of 20/1
[2] Composites were autoclave molded, for cure cycle see Table 16
[3] Composites were postcured at 477 K (204°C) for 24 hrs
[4] Samples failed in compression
[5] After 20 min soak at temperature

Table 33

Flexural Properties[1] of Humidity Exposed[2] Postcured Celion 6000/Epoxy Resin Composites[3,4]

| Composite No. | RT Strength[5] ksi | RT Strength[5] MPa | RT Modulus $10^6$psi | RT Modulus GPa | 423 K (150°C)[6] Strength[5] ksi | 423 K (150°C)[6] Strength[5] MPa | 423 K (150°C)[6] Modulus $10^6$psi | 423 K (150°C)[6] Modulus GPa | 450 K (177°C)[6] Strength[5] ksi | 450 K (177°C)[6] Strength[5] MPa | 450 K (177°C)[6] Modulus $10^6$psi | 450 K (177°C)[6] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1-C | 206 | 1420 | 19.7 | 136 | 181 | 1252 | 19.4 | 133 | 129[7] | 888 | 17.9 | 124 |
| | 215 | 1480 | 19.4 | 134 | 180 | 1243 | 19.2 | 132 | 129 | 891 | 17.9 | 124 |
| 2-IME-1 | 189 | 1302 | 18.1 | 125 | 94 | 647 | 17.1 | 118 | 88 | 604 | 14.2 | 98.1 |
| | 117 | 807 | 10.3 | 71.1 | 119 | 825 | 18.5 | 128 | 94 | 651 | 14.8 | 102 |
| 3-IME-2 | 146 | 1007 | 16.6 | 115 | 103 | 710 | 15.3 | 105 | 53 | 366 | 7.61 | 52.5 |
| | 170 | 1174 | 17.9 | 122 | 107 | 737 | 14.1 | 97.4 | 55 | 381 | 7.81 | 53.9 |
| 4-IME-3 | 170 | 1174 | 17.1 | 118 | 96 | 662 | 13.4 | 92.5 | 59 | 408 | 7.34 | 50.7 |
| | 156 | 1077 | 17.6 | 122 | | | | | | | | |
| 5-IME-4 | 196 | 1354 | 18.2 | 126 | 66.6 | 459 | 14.1 | 97.4 | 61.0 | 421 | 11.7 | 80.8 |
| | 186 | 1284 | 17.9 | 124 | 70.2 | 485 | 15.0 | 104 | 43.9 | 303 | 8.28 | 57.1 |
| 6-IME-5 | 227 | 1567 | 17.1 | 118 | 100 | 690 | 16.4 | 113 | 75.6 | 522 | 11.6 | 80.1 |
| | 240 | 1657 | 20.2 | 140 | 102 | 704 | 16.5 | 114 | 108 | 746 | 17.4 | 120 |
| 7-IME-6 | 186 | 1279 | 18.4 | 127 | 89.8 | 619 | 18.3 | 126 | 62.4 | 430 | 20.2 | 139 |
| | 147 | 1012 | 18.6 | 128 | 94.7 | 653 | 18.2 | 125 | 65.3 | 450 | 17.8 | 123 |
| 8-IME-7 | 190 | 1312 | 16.5 | 114 | 82.3 | 567 | 13.8 | 95.4 | 57.2 | 395 | 14.9 | 103 |
| | 195 | 1347 | 16.3 | 112 | 83.7 | 574 | 14.6 | 101 | 62.1 | 428 | 13.9 | 95.9 |
| 9-IME-8 | 197 | 1357 | 18.0 | 124 | 77.7 | 536 | 14.8 | 102 | 49.8 | 343 | 11.3 | 78.0 |
| | 202 | 1392 | 18.5 | 128 | 75.9 | 523 | 14.8 | 102 | 49.2 | 339 | 9.0 | 62.0 |

[1] Three point flexure at a span-to-depth ratio 20/1
[2] 87% RH @ 355 K (82°C) to saturation
[3] composites were autoclave molded, for cure cycle see Table 16
[4] composites were postcured at 477 K (204°C) for 24 hrs
[5] most samples failed in compression
[6] after 20 min soak at temperature
[7] failed in shear

Table 34

Flexural Properties of Celion 6000/IME Epoxy Resin Composites[1,2]
(Dry Condition)

| Composite No. | "as fabricated" RT Strength[3] ksi | RT Strength[3] MPa | RT Modulus $10^6$psi | RT Modulus GPa | 450 K (177°C)[7] Strength[3,4] ksi | 450 K Strength[3,4] MPa | 450 K Modulus $10^6$psi | 450 K Modulus GPa | 477 K (204°C)[7] Strength[3,4] ksi | 477 K Strength[3,4] MPa | 477 K Modulus $10^6$psi | 477 K Modulus GPa | Postcured 3 hrs at 477 K (204°C) 423 K (150°C)[7] Strength[5,6] ksi | Postcured Strength[5,6] MPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 11-IME-2 | 210 | 1447 | 15.0 | 103 | 125 | 860 | 13.9 | 96.2 | 73.9 | 510 | 12.1 | 83.7 | 129 | 891 |
| 13-IME-3 | 219 | 1513 | 16.0 | 110 | 77.9 | 537 | 12.8 | 88.4 | 33.9 | 234 | 5.70 | 39.3 | 85.0 | 585 |
| 14-IME-4 | 173 | 1196 | 15.2 | 105 | 37.1 | 256 | 6.54 | 45.1 | 31.9 | 220 | 2.63 | 18.1 | 87.6 | 604 |
| 15-IME-5 | 229 | 1581 | 15.5 | 107 | 88.8 | 612 | 14.6 | 101 | 51.8 | 357 | 9.59 | 66.1 | 103 | 707 |
| 17-IME-7 | 200 | 1382 | 15.6 | 108 | 46.5 | 321 | 8.14 | 56.1 | 99.4 | 685 | 9.72 | 67.0 | 79.3 | 547 |
| 18-IME-8 | 233 | 1605 | 17.5 | 121 | 58.9 | 406 | 12.7 | 87.9 | 118 | 816 | 10.9 | 75.1 | 72.1 | 497 |

[1] Composites (10 cm x 10 cm x ~0.254 cm) were compression molded, similar to the process described in Table 17 for larger composites
[2] Composites were not postcured
[3] Three point flex test at a span-to-depth ratio of 20/1, except where noted
[4] samples failed in compression
[5] Four point flexure test at a span-to-depth ratio of 20/1
[6] Samples failed in tension
[7] after 20 min soak at temperature

Table 35

Flexural Properties[1] of "As Fabricated", Celion 6000/Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | RT Strength ksi | RT Strength MPa | RT Modulus 10 psi | RT Modulus GPa | 450 K (177°C)[5] Strength ksi | 450 K (177°C)[5] Strength MPa | 450 K (177°C)[5] Modulus 10 psi | 450 K (177°C)[5] Modulus GPa | 477 K (204°C)[5] Strength ksi | 477 K (204°C)[5] Strength MPa | 477 K (204°C)[5] Modulus $10^3$psi | 477 K (204°C)[5] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 33-C | 278 | 1917 | 19.2 | 132 | 102 | 704 | 17.0 | 117 | 58.9 | 406 | 12.3 | 84.6 |
| | 229 | 1581 | 19.8 | 137 | 106 | 734 | 17.7 | 122 | 55.6 | 384 | 10.7 | 73.9 |
| 20-IME-1 | 250 | 1724 | 12.3 | 84.6 | 165 | 1140 | 13.2 | 90.9 | 65.4 | 451 | 11.2 | 77.3 |
| | 184[4] | 1267 | 16.5 | 114 | 102 | 704 | 12.5 | 85.9 | 69.7 | 481 | 9.37 | 64.6 |
| 32-IME-2 | 221 | 1523 | 17.2 | 119 | 25.0 | 172 | 4.33 | 29.9 | 26.3 | 181 | 3.95 | 27.2 |
| | 160 | 1100 | 19.0 | 131 | 26.2 | 180 | 17.0 | 117 | 28.1 | 194 | 4.37 | 30.1 |
| 28-IME-3 | 187 | 1290 | 17.9 | 123 | 23.1 | 159 | 3.93 | 27.1 | 31.0 | 214 | 4.05 | 28.0 |
| | 172 | 1180 | 16.8 | 116 | 27.8 | 192 | 4.34 | 29.9 | 29.0 | 200 | 4.40 | 30.4 |
| 29-IME-4 | 247 | 1701 | 16.8 | 116 | 71.7 | 494 | 13.2 | 90.8 | 40.7 | 280 | 6.61 | 45.5 |
| | 198 | 1368 | 17.5 | 120 | 73.7 | 504 | 11.5 | 79.4 | 45.2 | 312 | 8.55 | 58.9 |
| 24-IME-5 | 270 | 1861 | 17.2 | 119 | 63.7 | 439 | 11.2 | 77.2 | 35.6 | 246 | 7.34 | 50.6 |
| | 179[4] | 1234 | 18.0 | 124 | 78.1 | 539 | 11.5 | 79.5 | 35.4 | 244 | 7.08 | 48.8 |
| 25-IME-6 | 186 | 1279 | 16.3 | 113 | 48.8 | 337 | 9.94 | 68.6 | 21.3 | 147 | 4.86 | 33.5 |
| | 144 | 990 | 15.6 | 107 | 36.5 | 252 | 6.30 | 43.4 | 22.8 | 157 | 4.90 | 33.8 |
| 26-IME-7 | 207 | 1427 | 16.3 | 113 | 46.8 | 323 | 7.81 | 53.8 | 23.0 | 159 | 4.84 | 33.4 |
| | 202 | 1393 | 16.4 | 113 | 47.5 | 327 | 9.80 | 67.5 | 27.2 | 188 | 5.90 | 40.7 |
| 27-IME-8 | 227 | 1568 | 16.8 | 111 | 38.0 | 262 | 6.82 | 47.0 | 28.4 | 196 | 4.35 | 30.0 |
| | 161 | 1109 | 15.1 | 104 | 35.4 | 244 | 8.60 | 59.3 | 25.9 | 178 | 4.84 | 33.4 |

[1] Three point flex test at a span-to-depth ratio of 20/1, except where noted
[2] Composites were compression molded, for cure cycle see Table 17
[3] Composites were not postcured
[4] Four point flexure test at a span-to-depth ratio of 20/1
[5] after 20 min soak at temperature

Table 36

Flexural Properties[1] of Boiling Water Exposed "As Fabricated" Celion 6000/Epoxy Resin Composite[2,3]
(Flexural Properties after 72 hrs Boiling Water Exposure)

| Composite No. | RT Strength[4] ksi | RT Strength[4] MPa | RT Modulus $10^6$psi | RT Modulus GPa | 450 K (177°C)[5] Strength[4] ksi | 450 K (177°C)[5] Strength[4] MPa | 450 K (177°C)[5] Modulus $10^6$psi | 450 K (177°C)[5] Modulus GPa | 477 K (204°C)[5] Strength[4] ksi | 477 K (204°C)[5] Strength[4] MPa | 477 K (204°C)[5] Modulus $10^6$psi | 477 K (204°C)[5] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 33-C | 204 | 1409 | 16.3 | 112 | 74.5 | 513 | 9.2 | 63.3 | 28.8 | 199 | 5.91 | 40.7 |
| 20-IME-1 | 239 | 1647 | 14.9 | 103 | 86.5 | 596 | 10.5 | 72.5 | 60.5 | 417 | 9.04 | 62.3 |
| 32-IME-2 | 168 | 1156 | 14.8 | 102 | 36.8 | 254 | 4.3 | 29.6 | 30.0 | 207 | 3.59 | 24.8 |
| 28-IME-3 | 191 | 1314 | 14.4 | 99.5 | 36.6 | 252 | 4.8 | 33.1 | 26.9 | 185 | 3.80 | 26.2 |
| 29-IME-4 | 222 | 1534 | 14.1 | 97.3 | 65.1 | 449 | 8.66 | 59.7 | 29.4 | 203 | 5.08 | 35.0 |
| 24-IME-5 | 240 | 1657 | 14.5 | 99.7 | 48.6 | 335 | 8.8 | 60.6 | 34.5 | 238 | 5.94 | 41.0 |
| 25-IME-6 | 133 | 918 | 13.8 | 95.2 | 57.3 | 395 | 8.8 | 55.6 | 30.6 | 211 | 4.68 | 32.3 |
| 26-IME-7 | 202 | 1394 | 15.0 | 103 | 61.0 | 420 | 8.4 | 58.1 | 27.7 | 191 | 6.22 | 42.9 |
| 27-IME-8 | 170 | 1169 | 14.2 | 98.0 | 38.4 | 264 | 5.6 | 38.6 | 25.5 | 176 | 5.07 | 35.0 |

[1] Three point flexure test at a span-to-depth ratio of 20/1
[2] Composites were compression molded, for cure cycle see Table 17
[3] Composites were not postcured
[4] Samples failed in compression
[5] after 20 min soak at temperature

Table 37

Flexural Properties[1] of Celion 6000/Epoxy Resin Composites[2]
(Dry Condition)

| Composite No. | Postcured 3 hrs at 477 K (204°C) — 423 K (150°C)[5] Strength | | Modulus | |
|---|---|---|---|---|
| | ksi | MPa | $10^6$psi | GPa |
| 33-C | 145[3] | 1005 | 8.9 | 61.3 |
| 20-IME-1 | 134[3] | 926 | – | – |
| 34-IME-1 | 105[3] | 724 | 15.7 | 108 |
| 32-IME-2 | 94.5[3] | 307 | 1.12 | 7.72 |
| 28-IME-3 | 73.1[4] | 504 | 14.2 | 98.2 |
| 23-IME-4 | 121[3] | 836 | 16.1 | 111 |
| 29-IME-4 | 111[4] | 768 | 14.6 | 102 |
| 24-IME-5 | 131[3] | 903 | 16.1 | 111 |
| 30-IME-5 | 131[4] | 903 | 18.6 | 126 |
| 25-IME-6 | 85.5[4] | 589 | 14.7 | 101 |
| 31-IME-6 | 74.1[3] | 510 | 14.9 | 103 |
| 26-IME-7 | 77.0[4] | 531 | 13.2 | 91.1 |

[1] Three point flex tests at a span-to-depth ratio of 20/1
[2] Composites were compression molded, for cure cycle see Table 17
[3] Samples failed in tension
[4] Samples failed in compression
[5] After 20 min soak at temperature

Table 38

Flexural Properties[1] of Celion 6000/Epoxy Resin Composites[2,3]
(Dry Condition)

| Composite No. | RT Strength ksi | RT Strength MPa | RT Modulus $10^6$psi | RT Modulus GPa | 423 K (150°C)[6] Strength ksi | 423 K (150°C)[6] Strength MPa | 423 K (150°C)[6] Modulus $10^6$psi | 423 K (150°C)[6] Modulus GPa | 450 K (177°C)[6] Strength ksi | 450 K (177°C)[6] Strength MPa | 450 K (177°C)[6] Modulus $10^6$psi | 450 K (177°C)[6] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 33-C | 248 | 1708 | 15.9 | 110 | 160 | 1106 | 15.4 | 106 | 120 | 829 | 16.6 | 114 |
| 20-IME-1 | 248[4] | 1710 | 16.9 | 116 | 159[4] | 1093 | 14.9 | 103 | 127[4] | 872 | 14.4 | 99.2 |
| | | | | | 140 | 968 | 16.1 | 111 | 129 | 889 | 15.0 | 103 |
| 34-IME-1 | 221[4] | 1520 | 16.2 | 112 | 163[4] | 1123 | 16.4 | 113 | 162[4] | 1117 | 15.8 | 109 |
| | 222 | 1528 | 16.4 | 113 | | | | | | | | |
| 32-IME-2 | 196 | 1349 | 15.1 | 104 | 74.5 | 514 | 14.1 | 96.9 | 40.9 | 282 | 8.76 | 60.4 |
| | | | | | 75.2 | 518 | 14.8 | 99.2 | 45.9 | 317 | 10.5 | 72.6 |
| 36-IME-2 | 198[4] | 1362 | 17.4 | 120 | 134[4] | 925 | 15.7 | 108 | 102[4] | 701 | 14.3 | 98.4 |
| 28-IME-3 | 224 | 1545 | 15.4 | 106 | 60.6 | 418 | 14.6 | 100 | 54.0 | 372 | 11.6 | 79.8 |
| | | | | | 104[4] | 714 | 14.3 | 98.4 | 93.1[4] | 642 | 13.3 | 91.5 |
| 29-IME-4 | 218 | 1504 | 14.1 | 97.3 | 113 | 781 | 14.9 | 103 | 98.1 | 676 | 14.4 | 99.2 |
| | | | | | 113 | 781 | 13.6 | 93.4 | 86.8 | 599 | 14.4 | 99.6 |
| 24-IME-5 | 246 | 1699 | 17.9 | 123 | 183 | 1260 | 17.1 | 118 | | | | |
| 30-IME-5 | 211 | 1452 | 20.1 | 139 | 191 | 1319 | 16.3 | 148 | 170 | 1171 | 18.4 | 127 |
| | 280 | 1928 | 16.3 | 113 | 176 | 1213 | 21.5 | 123 | 161 | 1107 | 16.7 | 115 |
| 31-IME-6 | 219 | 1508 | 14.6 | 101 | 103 | 708 | 14.8 | 102 | 54.2 | 374 | 11.7 | 80.7 |
| | 224 | 1547 | 17.2 | 119 | 110[4] | 756 | 14.6 | 100 | 98.2[4] | 677 | 15.4 | 106 |
| 26-IME-7 | 223 | 1539 | 16.9 | 117 | 87.6 | 604 | 12.8 | 88.3 | 69.8 | 482 | 13.2 | 90.7 |
| 35-IME-7 | 208[4] | 1435 | 17.7 | 122[4] | 133[4] | 918 | 15.9 | 110 | 102[4] | 703 | 14.5 | 100 |
| 37-IME-7 | 192[4] | 1323 | 16.6 | 114[4] | 115[4] | 795 | 14.2 | 97.9 | 100[4] | 692 | 13.9 | 96.0 |
| 27-IME-8 | 199 | 1369 | 14.6 | 100 | 119 | 823 | 13.9 | 96.1 | 91.8 | 633 | 13.1 | 90.5 |
| | 207 | 1430 | 14.9 | | 130 | 899 | 14.1 | 97.0 | 91.6 | 632 | 13.2 | 91.2 |
| 38-IME-8 | 66.5[5] | 459 | 12.8 | 88.5 | 97.4[5] | 672 | 14.8 | 102 | 96.8[4] | 668 | 14.3 | 98.8 |
| | 32.4[5] | 223 | 14.4 | 99.3 | 100[5] | 692 | 14.2 | 97.7 | 102[5] | 702 | 14.5 | 99.7 |

[1] Three point flexure test at a span-to-depth ratio of 20/1
[2] Composites were compression molded, for cure cycle see Table 17
[3] Composites were postcured at 477 K (204°C) for 24 hrs
[4] Samples failed in compression
[5] Samples failed in shear
[6] After 20 min soak at temperature

Table 39

Flexural Properties[1] of Humidity Exposed[2] Postcured Celion 6000/Epoxy Resin Composites[3,4]

| Composite No. | RT Strength[5] ksi | RT Strength[5] MPa | RT Modulus $10^6$psi | RT Modulus GPa | 423 K (150°C)[7] Strength[5] ksi | 423 K (150°C)[7] Strength[5] MPa | 423 K (150°C)[7] Modulus $10^6$psi | 423 K (150°C)[7] Modulus GPa | 450 K (177°C)[7] Strength[5] ksi | 450 K (177°C)[7] Strength[5] MPa | 450 K (177°C)[7] Modulus $10^6$psi | 450 K (177°C)[7] Modulus GPa |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 33-C | 216 | 1489 | 16.4 | 113 | | | | | 106 | 734 | 14.6 | 101 |
| 20-IME-1 | 226 | 1560 | 16.2 | 112 | 152 | 1050 | 14.7 | 102 | 110 | 762 | 12.7 | 87.7 |
| | 236 | 1628 | 16.2 | 112 | 144 | 993 | 13.2 | 91.1 | 108 | 746 | 13.2 | 91.1 |
| 32-IME-2 | 225 | 1549 | 17.5 | 121 | 100 | 692 | 14.8 | 102 | 51 | 357 | 9.10 | 62.8 |
| | 207 | 1427 | 16.8 | 116 | 95 | 657 | 16.1 | 111 | 52 | 384 | 8.90 | 61.4 |
| 28-IME-3 | 202 | 1398 | 16.2 | 112 | 134 | 928 | 15.8 | 109 | 52 | 356 | 7.06 | 48.7 |
| | 214 | 1477 | 17.3 | 119 | | | | | 55 | 383 | 7.20 | 49.7 |
| 29-IME-4 | 213 | 1468 | 15.9 | 110 | 106 | 733 | 14.9 | 103 | 88 | 606 | 13.0 | 89.7 |
| | 222 | 1530 | 16.1 | 111 | 104 | 715 | 13.5 | 93.2 | 91 | 625 | 12.6 | 87.0 |
| 24-IME-5 | 210 | 1448 | 16.8 | 114 | 152 | 1051 | 15.6 | 107 | | | | |
| 30-IME-5 | 239 | 1650 | 15.6 | 107 | 141 | 973 | 15.6 | 107 | 124 | 855 | 14.6 | 100 |
| | | | | | 140 | 962 | 15.0 | 103 | | | | |
| 31-IME-6 | 222 | 1531 | 18.6 | 128 | 124 | 854 | 14.7 | 102 | 79 | 544 | 10.8 | 74.6 |
| | 234 | 1615 | 17.6 | 122 | 113 | 778 | 13.5 | 93.2 | 108 | 744 | 14.5 | 100 |
| 26-IME-7 | 165 | 1136 | 15.2 | 105 | 116 | 803 | 13.3 | 91.8 | 74.7 | 515 | 11.9 | 82.1 |

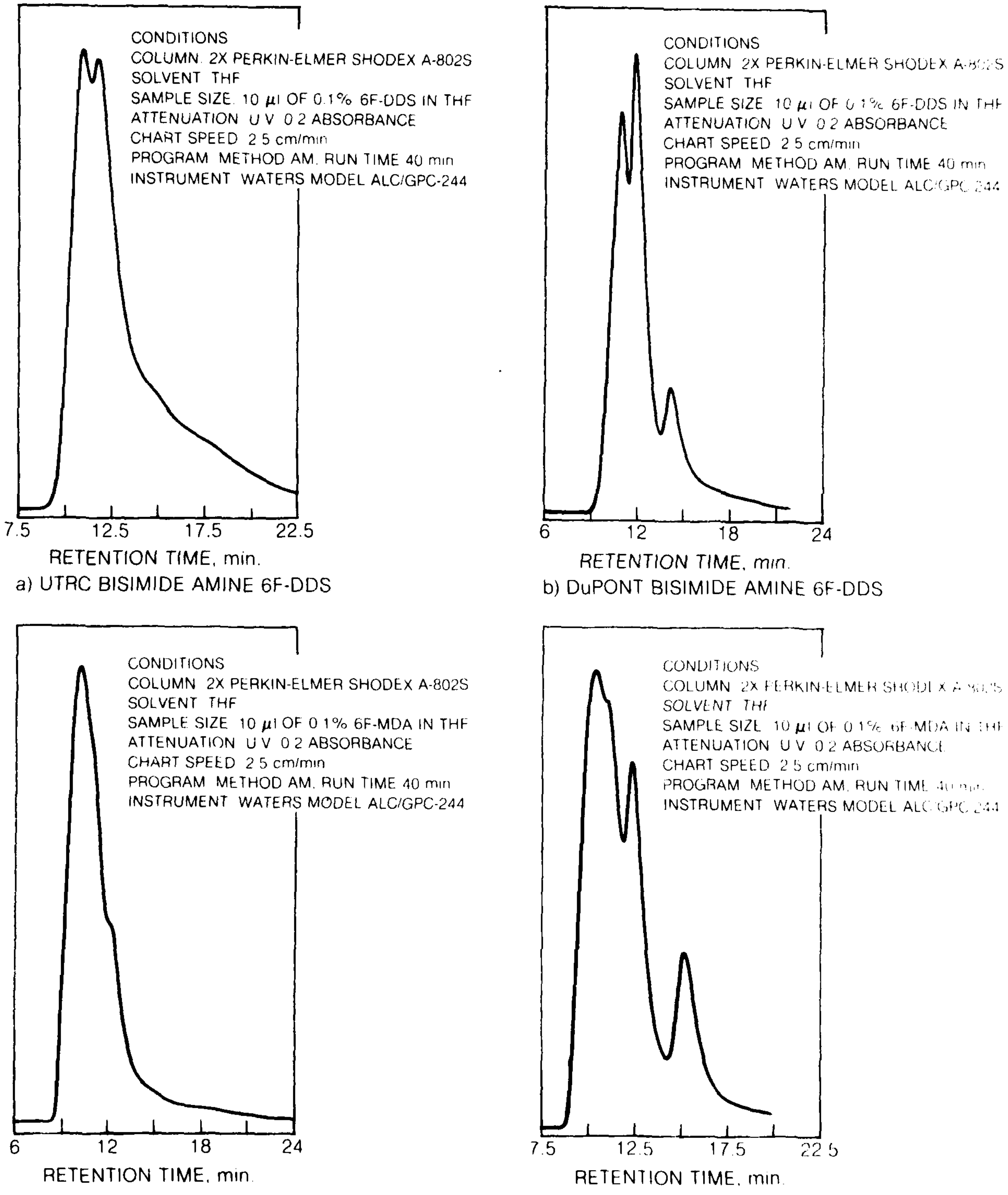
Table 39 (Cont'd)

| Composite No. | RT | | | | 423 K (150°C)[7] | | | | 450 K (177°C)[7] | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Strength | | Modulus | | Strength | | Modulus | | Strength | | Modulus | |
| | ksi | MPa | $10^6$psi | GPa | ksi | MPa | $10^6$psi | GPa | ksi | MPa | $10^6$psi | GPa |
| 27-IME-8 | 214 | 1452 | 16.5 | 114 | 84.6 | 583 | 15.6 | 107 | 90.7 | 625 | 12.2 | 84.2 |
| | 168 | 1160 | 14.3 | 98.7 | 84.4 | 502 | 13.2 | 80.9 | | | | |
| 38-IME-8 | 88 | 605 | 15.1 | 104 | 58 | 402 | 10.7 | 73.9 | 47[6] | 325 | 9.51 | 65.7 |
| | 72 | 500 | 15.2 | 105 | 51[6] | 351 | 9.7 | 66.9 | 46 | 314 | 8.49 | 58.7 |

1 Three point flex test at a span-to-depth of 20/1
2 87% RH at 355 K (82°C) to saturation
3 composites were compression molded composites; for cure cycle see Table 17
4 composites were postcured at 204°C for 24 hrs
5 most samples failed in compression
6 failed in shear
7 after 20 min soak at temperature

FIG. 1

## GEL PERMEATION CHROMATOGRAM OF BISIMIDE AMINES




a) UTRC BISIMIDE AMINE 6F-DDS

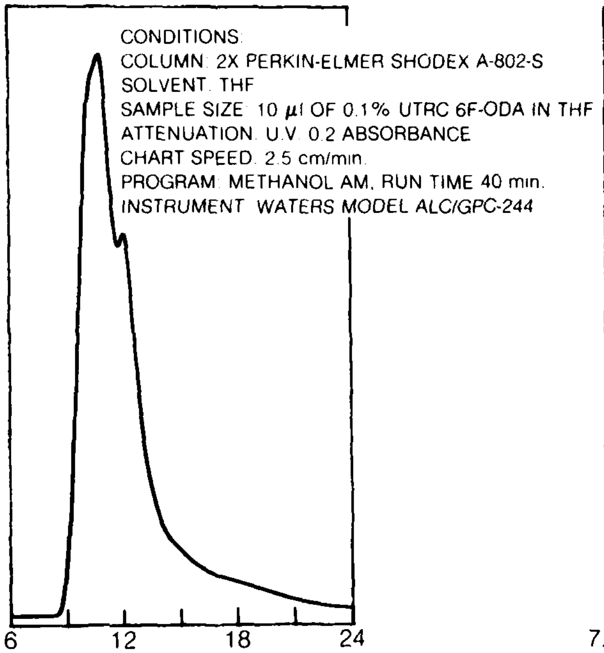
b) DuPONT BISIMIDE AMINE 6F-DDS

c) UTRC BISIMIDE AMINE 6F-MDA

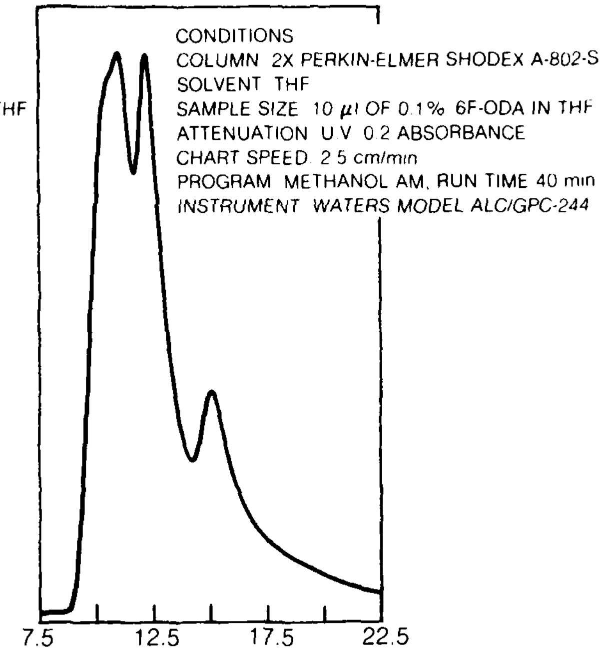
d) DuPONT BISIMIDE AMINE 6F-MDA

FIG. 2

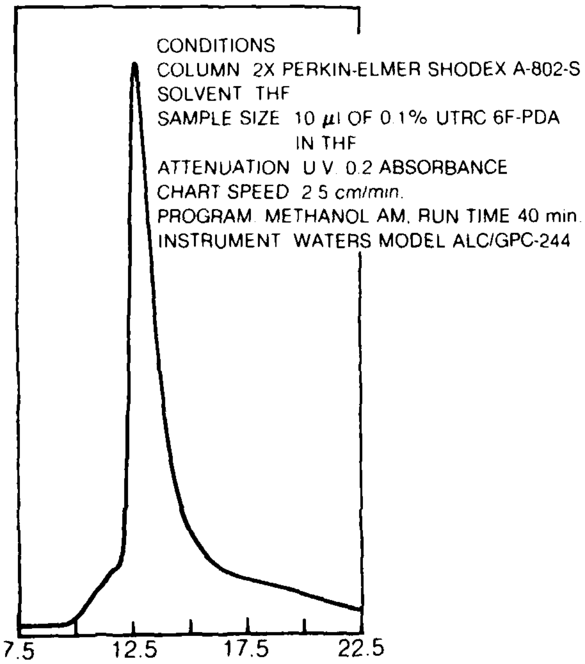
# GEL PERMEATION CHROMATOGRAM OF BISIMIDE AMINES



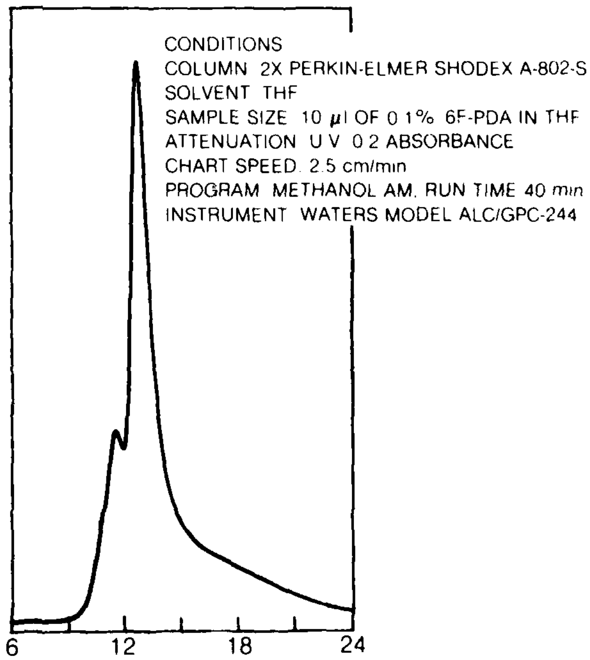

a) UTRC BISIMIDE AMINE 6F-ODA




b) DuPONT BISIMIDE AMINE 6F-ODA




c) UTRC BISIMIDE AMINE 6F-PDA

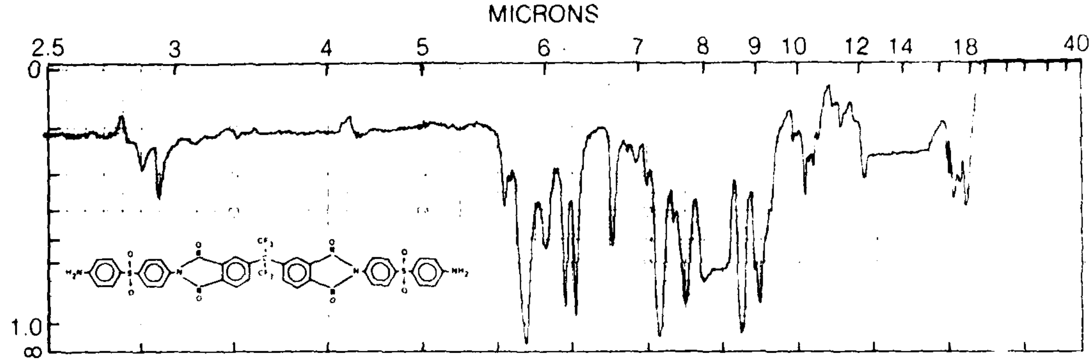



d) DuPONT BISIMIDE AMINE 6F-PDA

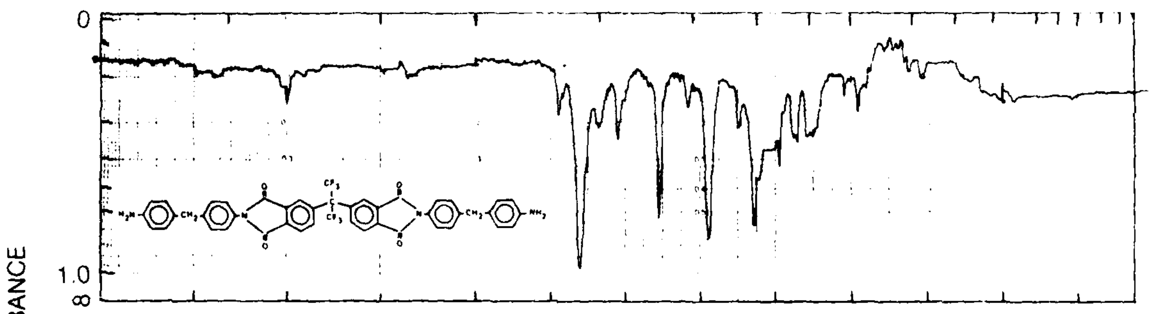
80–10–159–25

FIG. 3

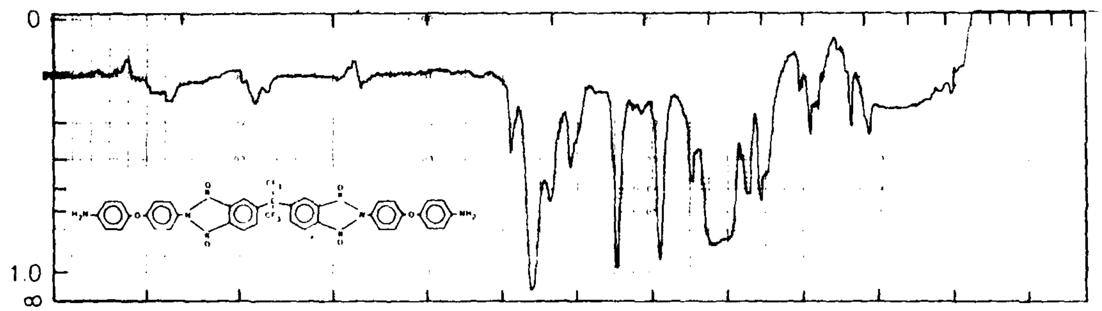
## INFRAFED SPECTRUM OF BISIMIDE AMINES



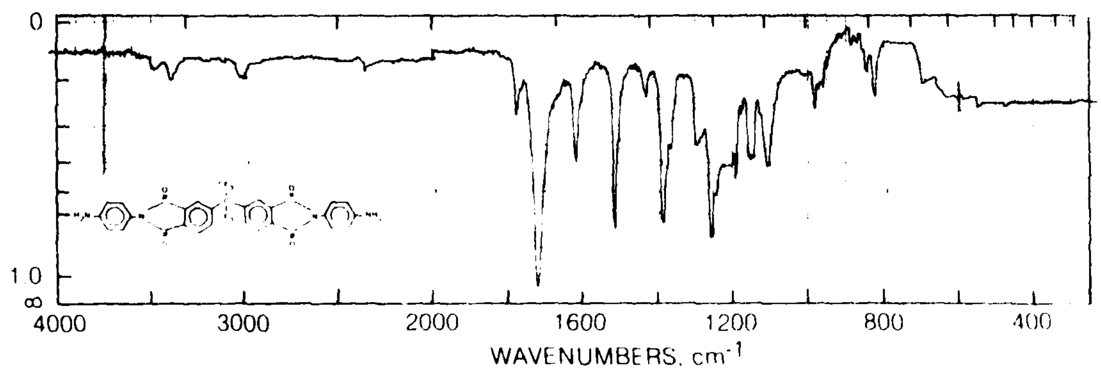

a) BISIMIDE AMINE 6F-DDS




b) BISIMIDE AMINE 6F-MDA



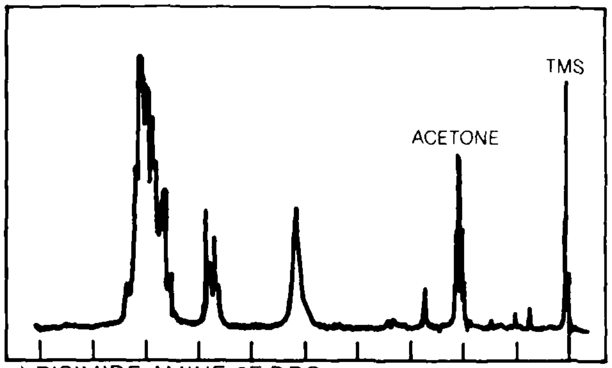

c) BISIMIDE AMINE 6F-ODA




d) BISIMIDE AMINE 6F-PDA

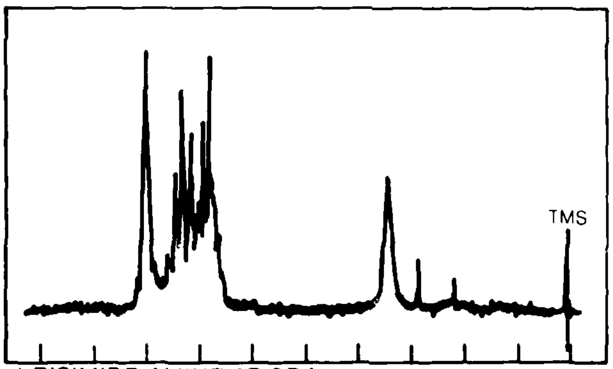
FIG. 4

## NMR SPECTRUM OF BISIMIDE AMINES

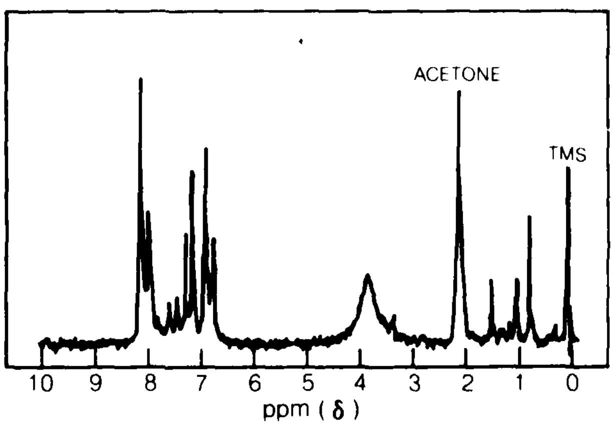



a) BISIMIDE AMINE 6F-DDS


b) BISIMIDE AMINE 6F-MDA




c) BISIMIDE AMINE 6F-ODA




d) BISIMIDE AMINE 6F-PDA

80-10-159-12

## DSC CHROMATOGRAM OF BISIMIDE AMINES

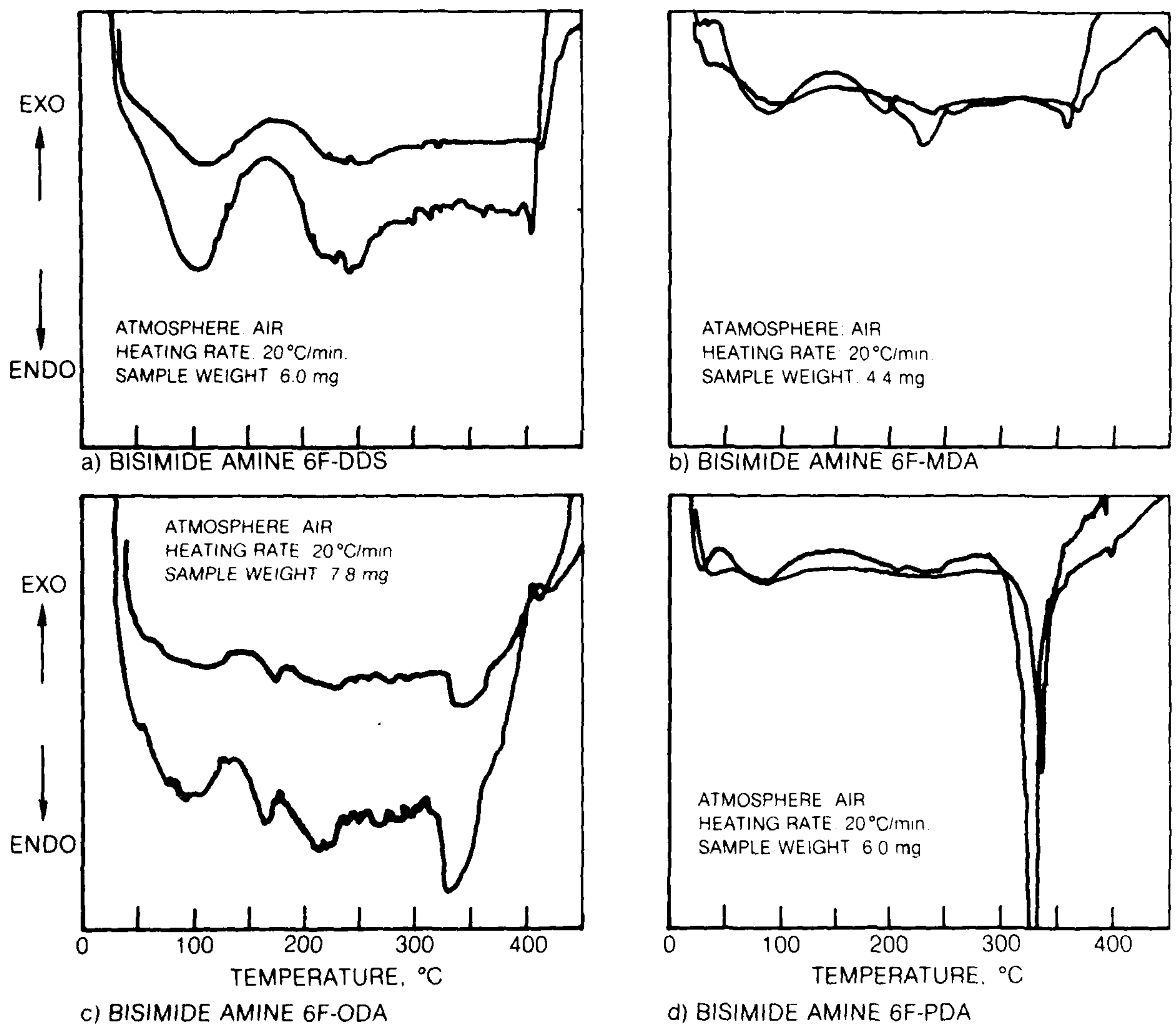
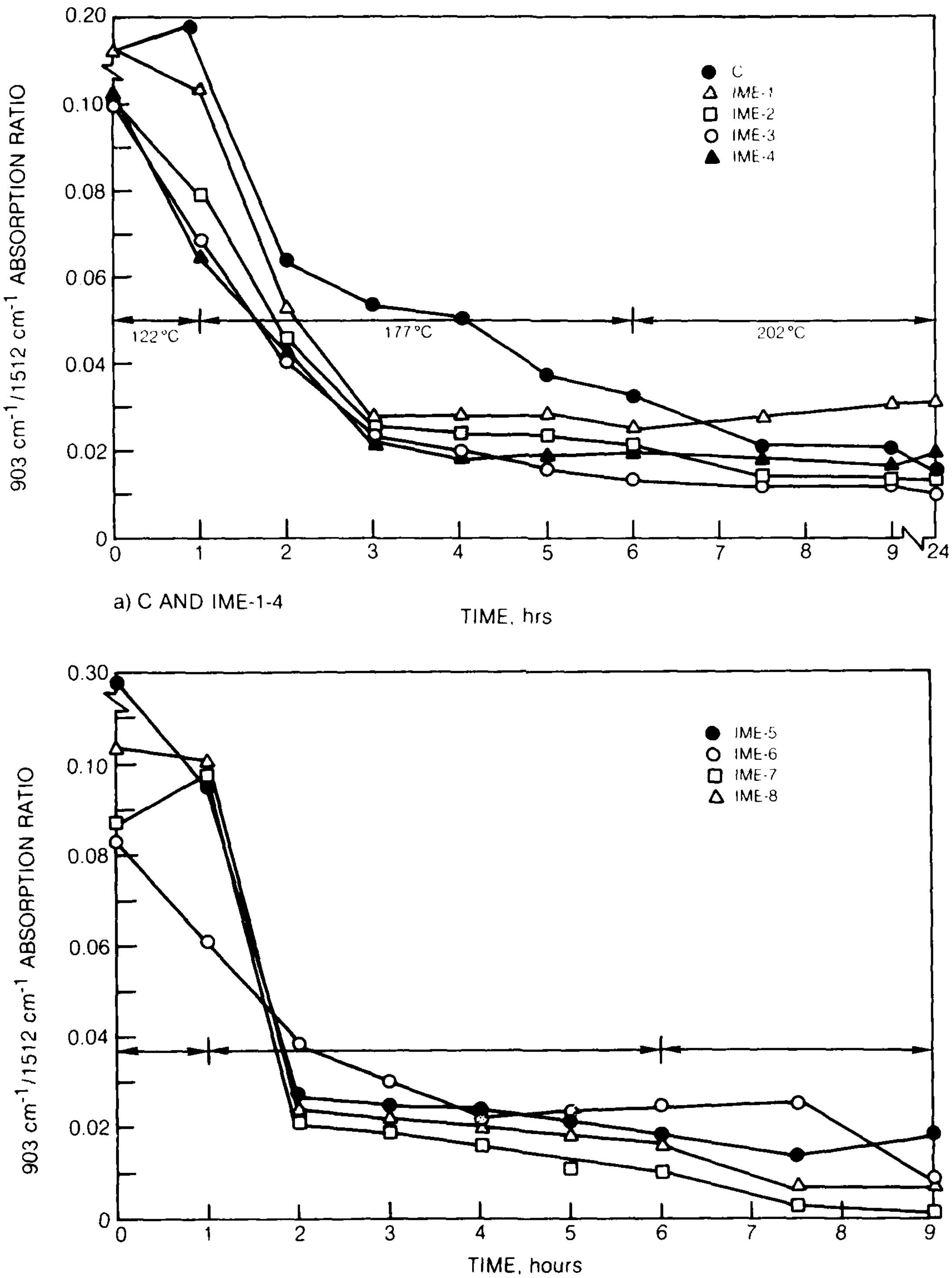


FIG. 6

**IR CURE STUDY OF EPOXY RESINS**

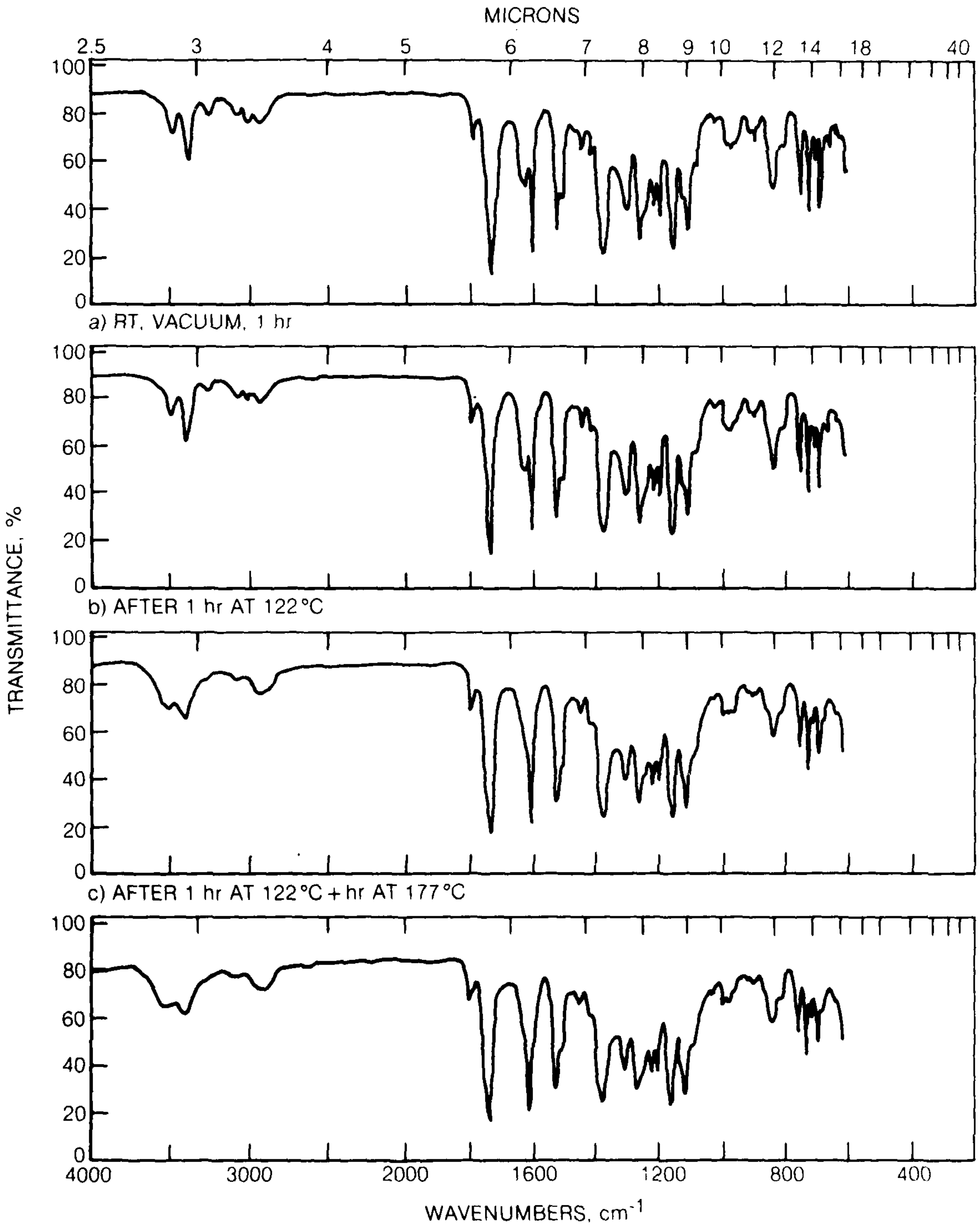



a) C AND IME-1-4

b) IME-5-8

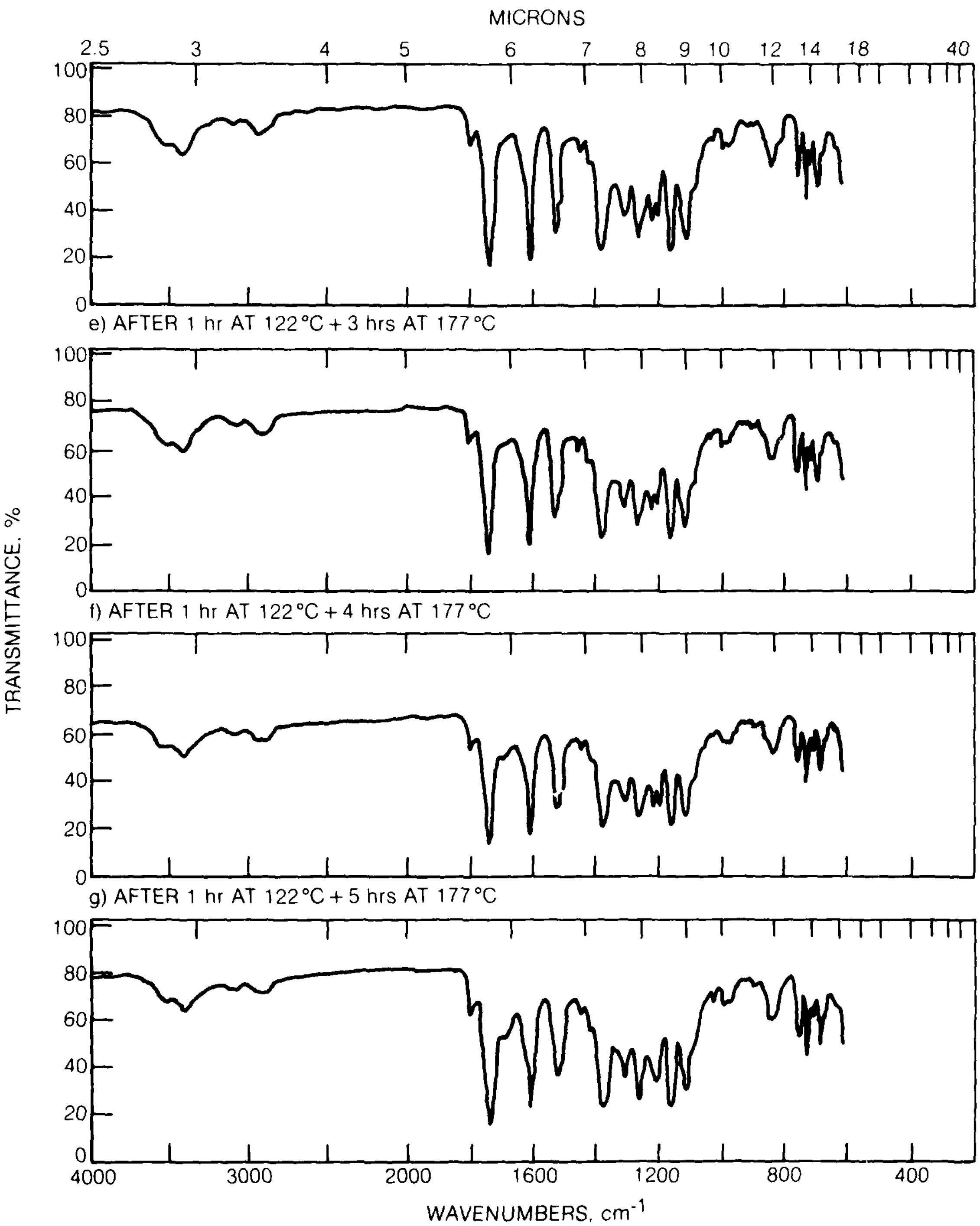
80–10–159–27

## IR OF IME-1 EPOXY RESIN CURE PROCESS




d) AFTER 1 hr AT 122°C + 2 hrs AT 177°C

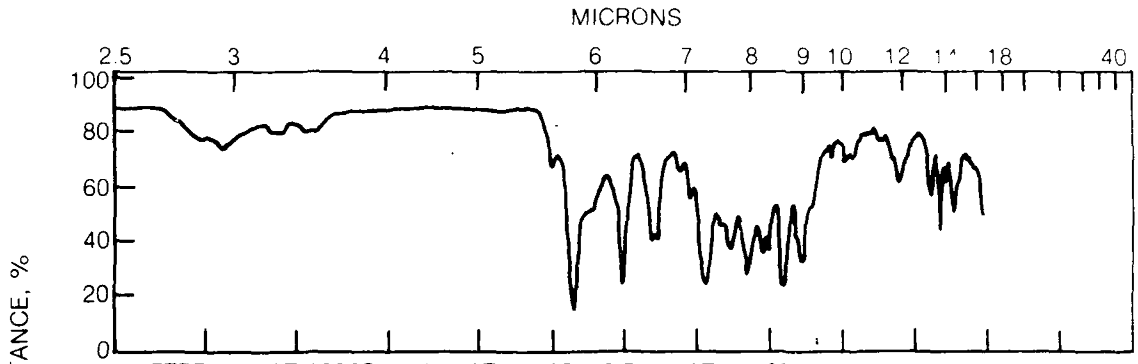
**IR OF IME-1 EPOXY RESIN CURE PROCESS (CONTINUED)**



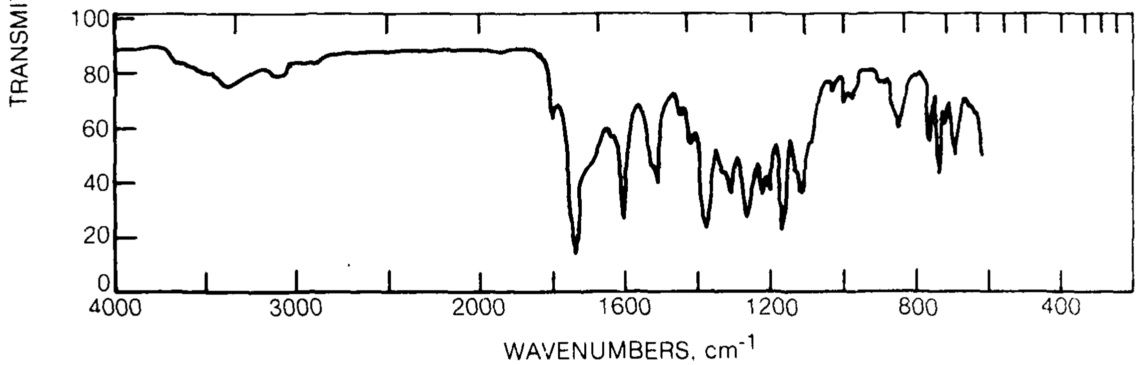

h) AFTER 1 hr AT 122°C + 5 hrs AT 177°C + 1.5 hrs AT 202°C

80-10-159-33

## IR OF IME-1 EPOXY RESIN CURE PROCESS (CONCLUDED)



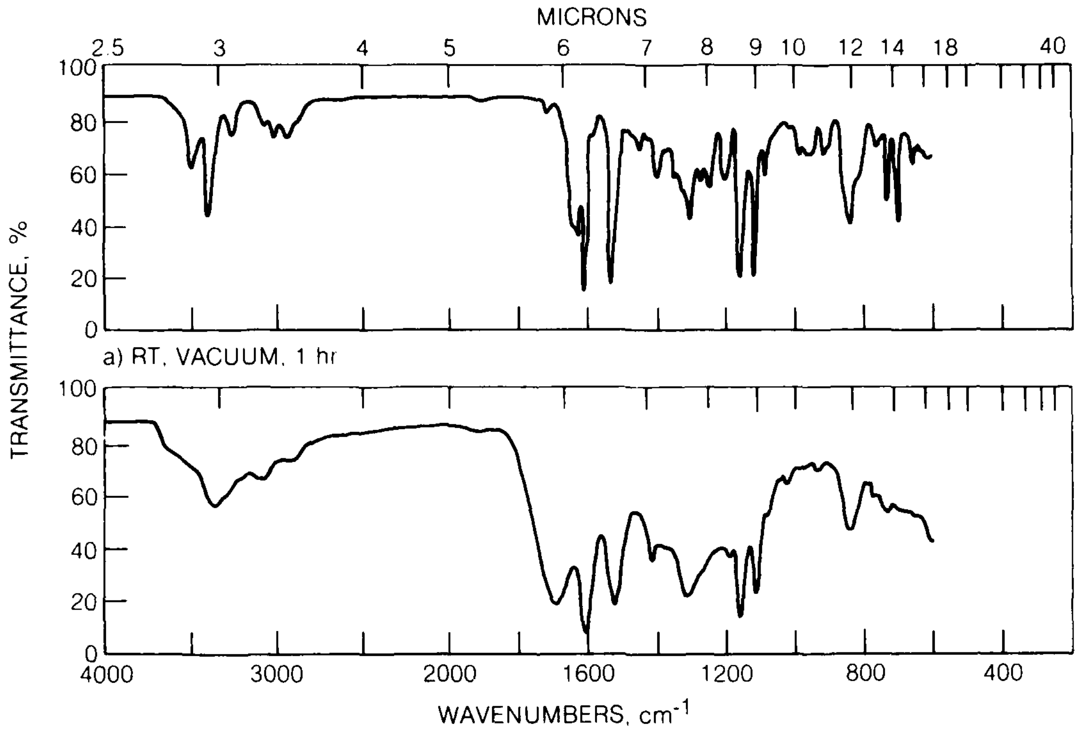

i) AFTER 1 hr AT 122 °C + 5 hrs AT 177 °C + 2.5 hrs AT 202 °C




j) AFTER 1 hr AT 122 °C + 5 hrs AT 177 °C + 24 hrs AT 202 °C

FIG. 8

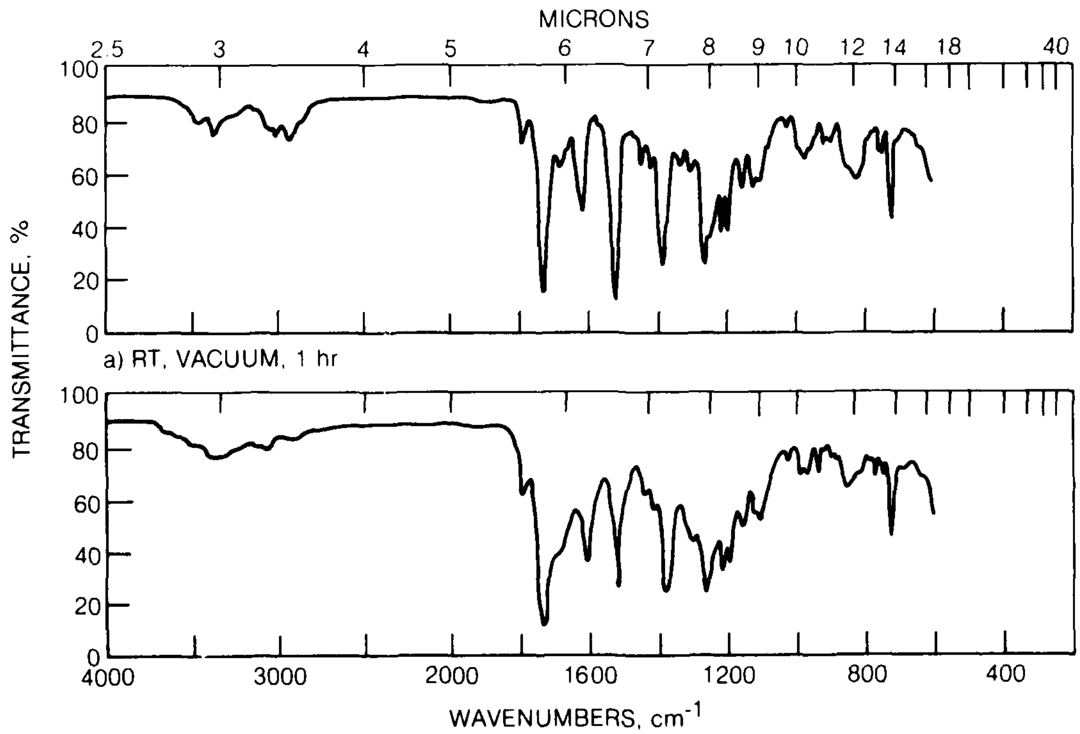
## IR OF CONTROL RESIN




b) 1 hr AT 122°C + 5 hr AT 177°C + 24 hrs AT 202°C

## IR OF IME-2

FIG. 9




b) 1 hr AT 122°C + 5 hrs AT 177°C + 24 hrs AT 202°C

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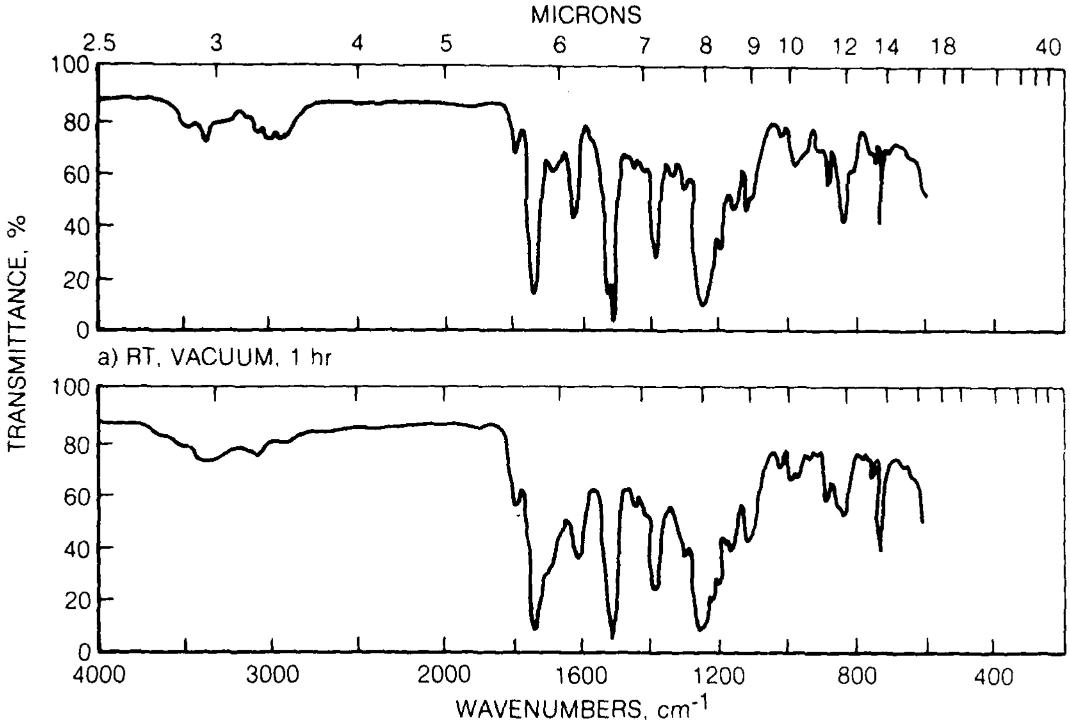
FIG. 10

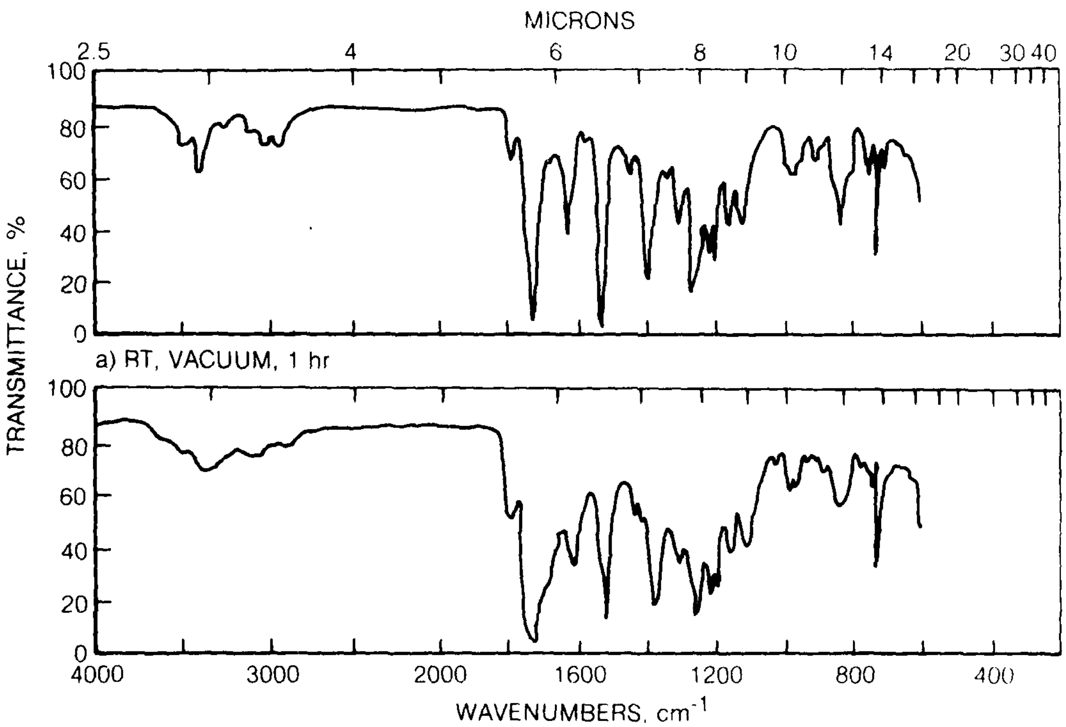
## IR OF IME-3 RESIN




b) 1 hr at 122°K + 5 hrs AT 177°C + 24 hrs at 202°C

## IR OF IME-4 RESIN

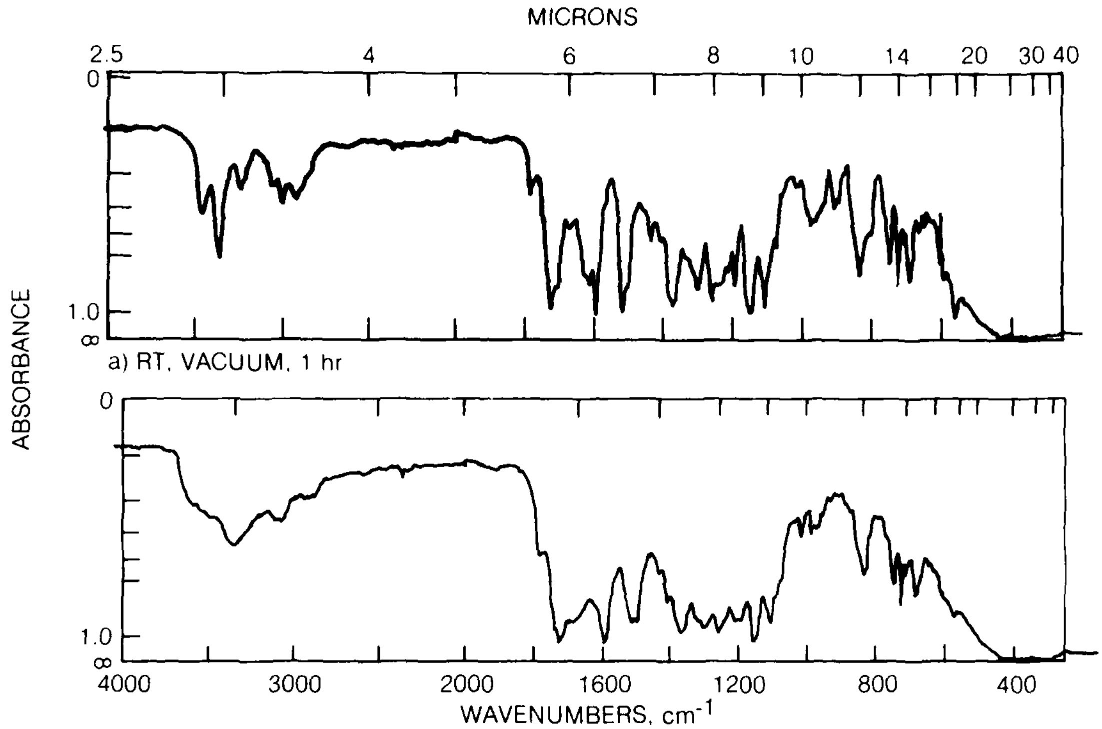
FIG. 11




b) 1 hr AT 122°K + 5 hrs AT 177°C + 24 hrs AT 202°C

FIG. 12

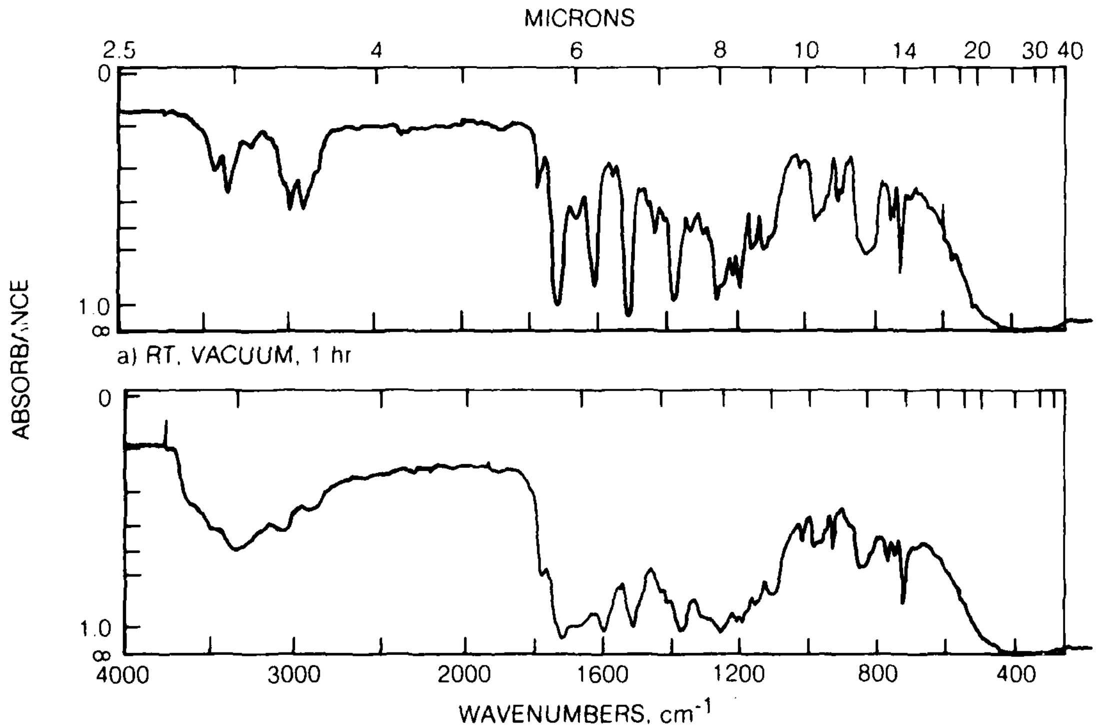
**IR OF IME-5 RESIN**




b) 1 hr AT 122°C, 5 hr AT 177°C, 2.5 hr AT 202°C

FIG. 13

**IR OF IME-6 RESIN**

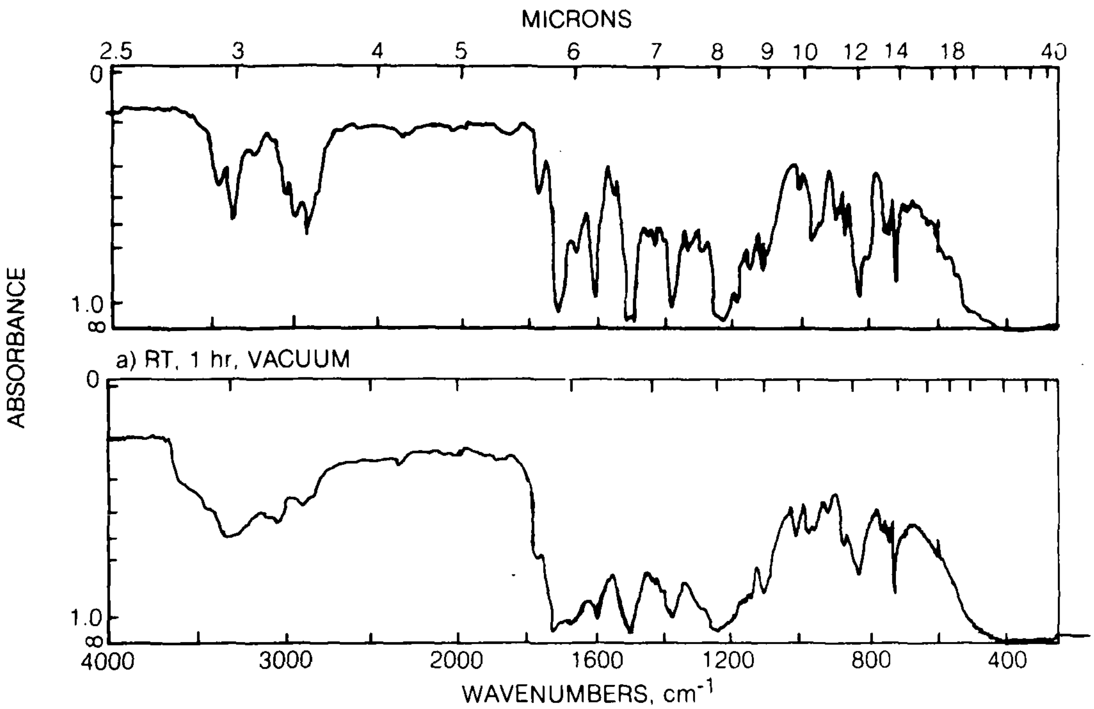



b) 1 hr AT 122°C, 5 hr AT 177°C, 2.5 hr AT 202°C

80–10–159–18

## IR OF IME-7

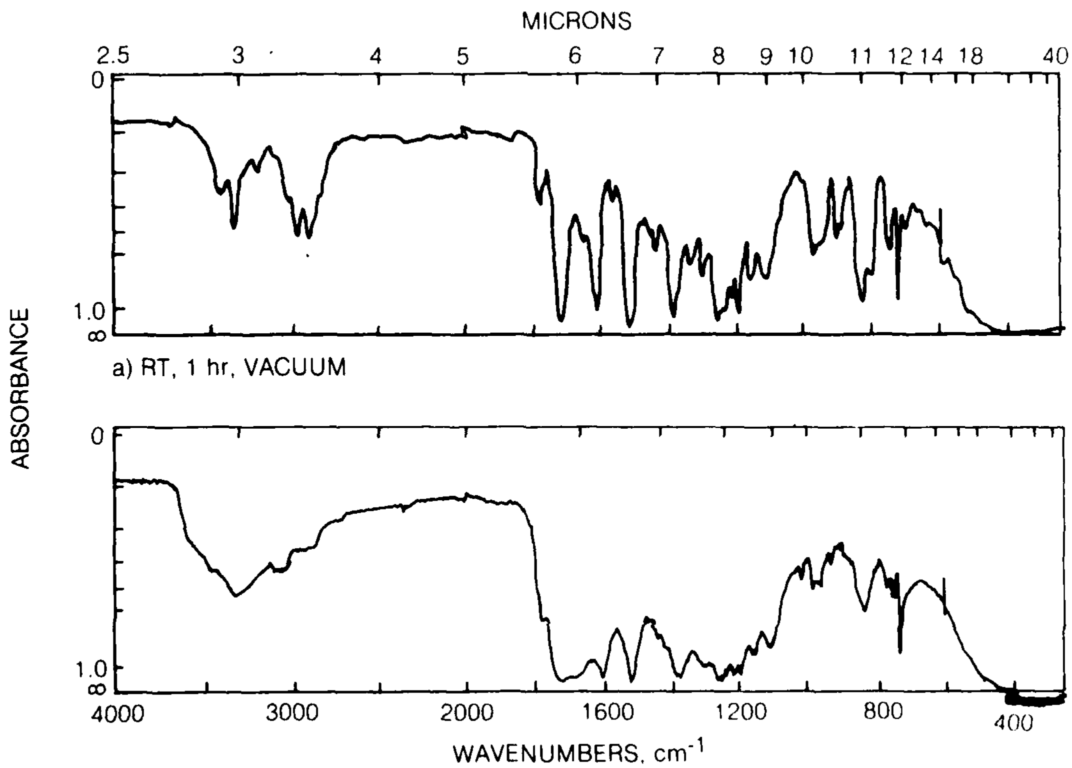
FIG. 14




b) 1 hr AT 122 °C + 5 hrs AT 177 °C + 2.5 hrs AT 202 °C

## IR OF IME-8

FIG 15

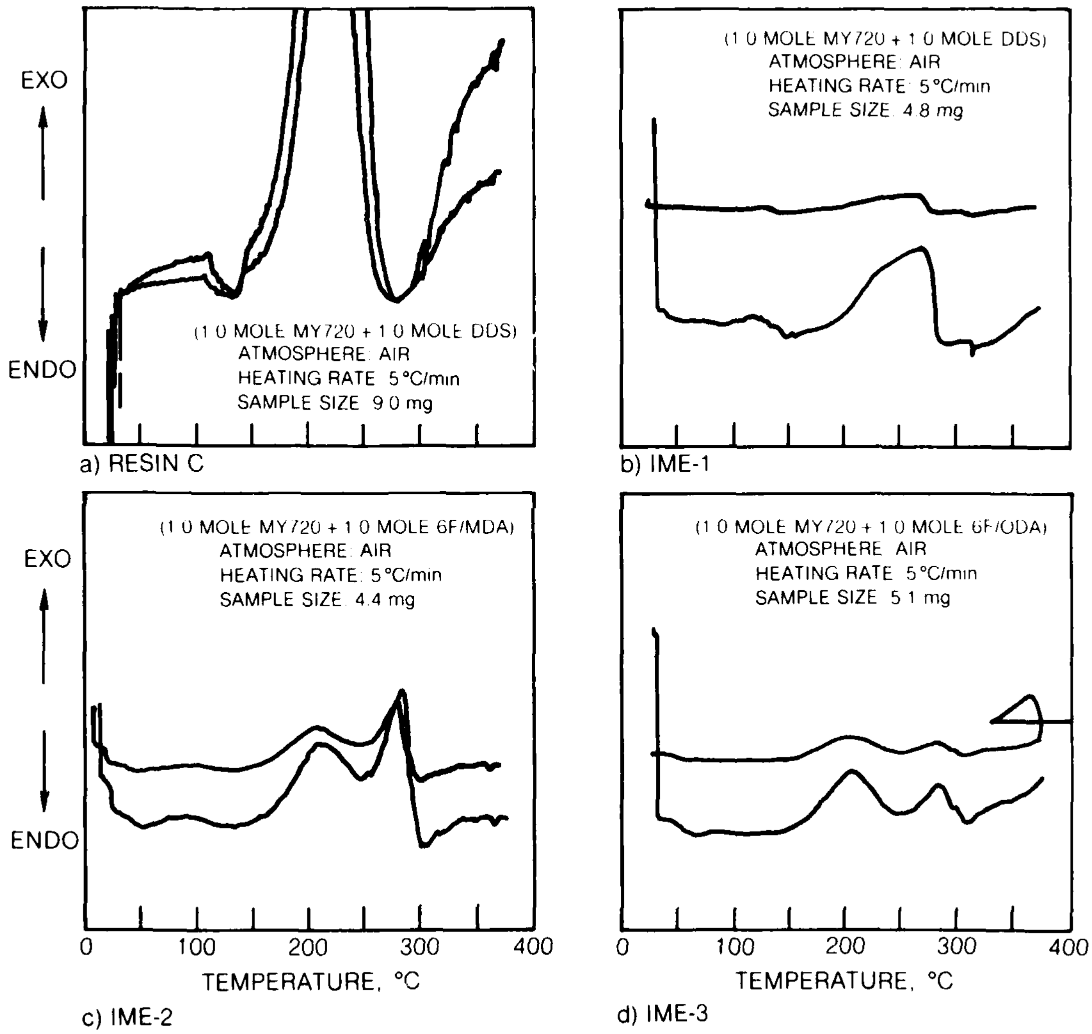



b) 1 hr AT 122 °C + 5 hrs AT 177 °C + 2.5 hrs AT 202 °C

80–10 159 29

FIG. 16

## DSC THERMOGRAMS OF UNCURED EPOXY RESINS




80-10-159-15

**DSC THERMOGRAMS OF UNCURED EPOXY RESINS (CONTINUED)**

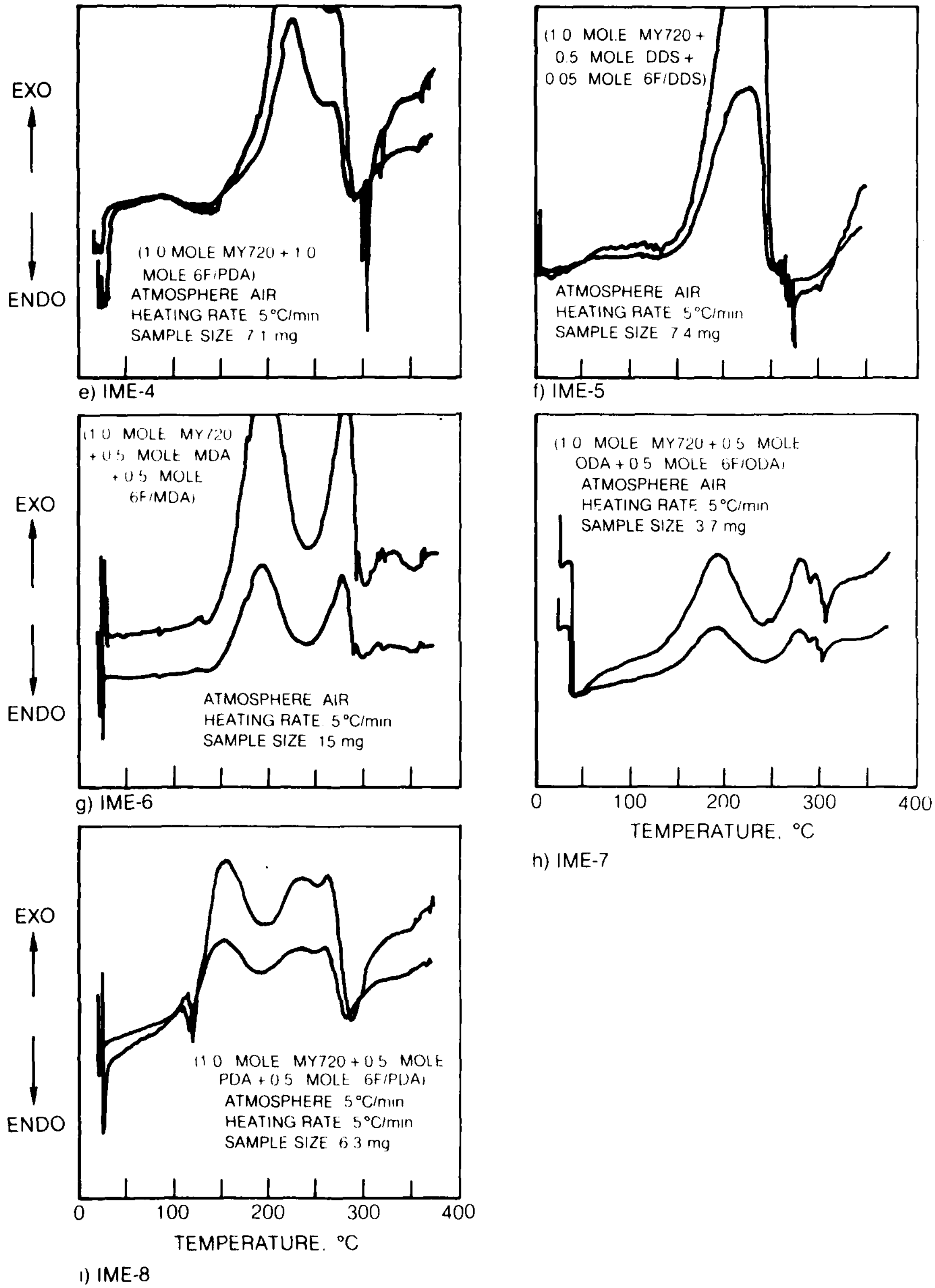
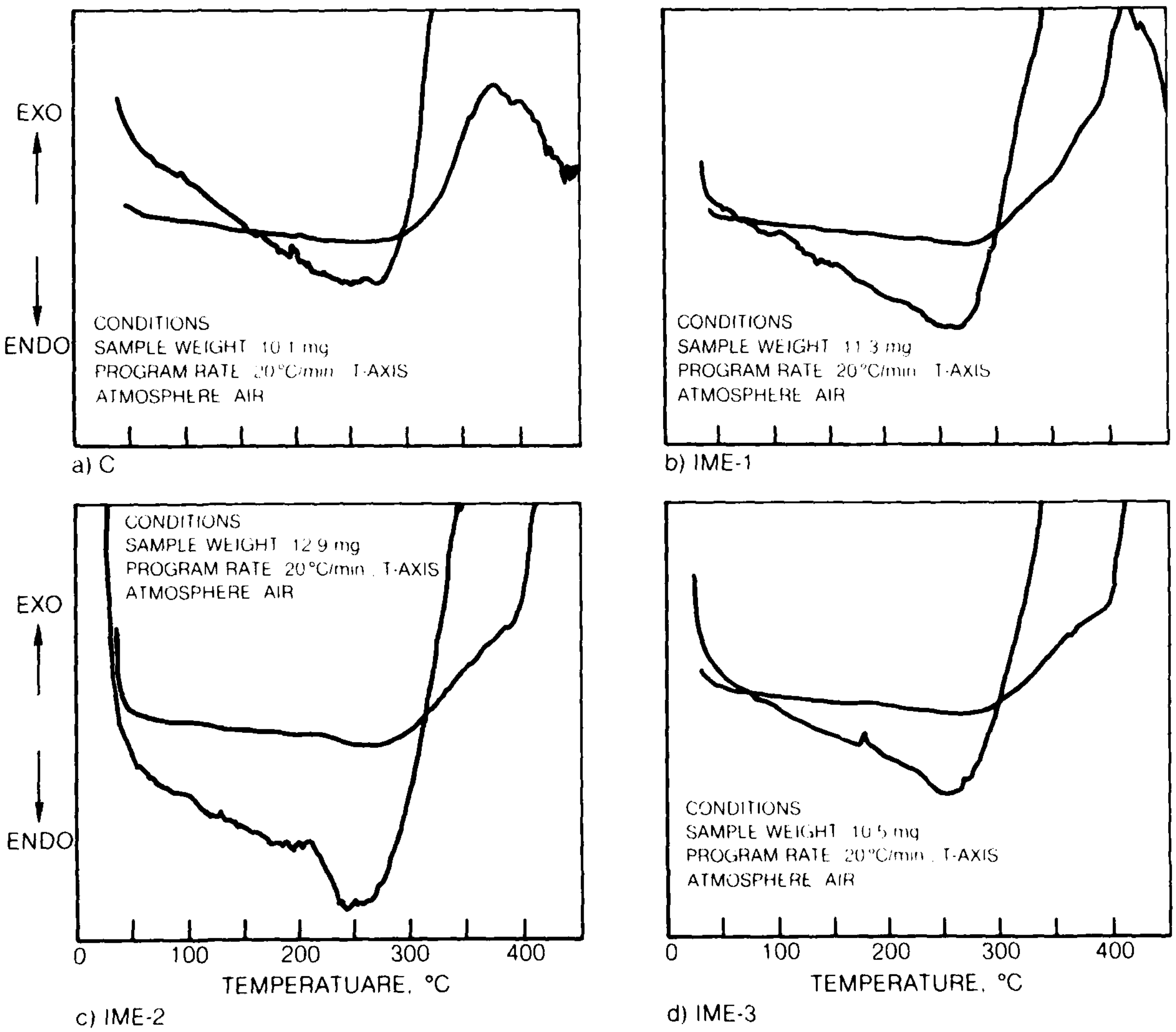


FIG. 17

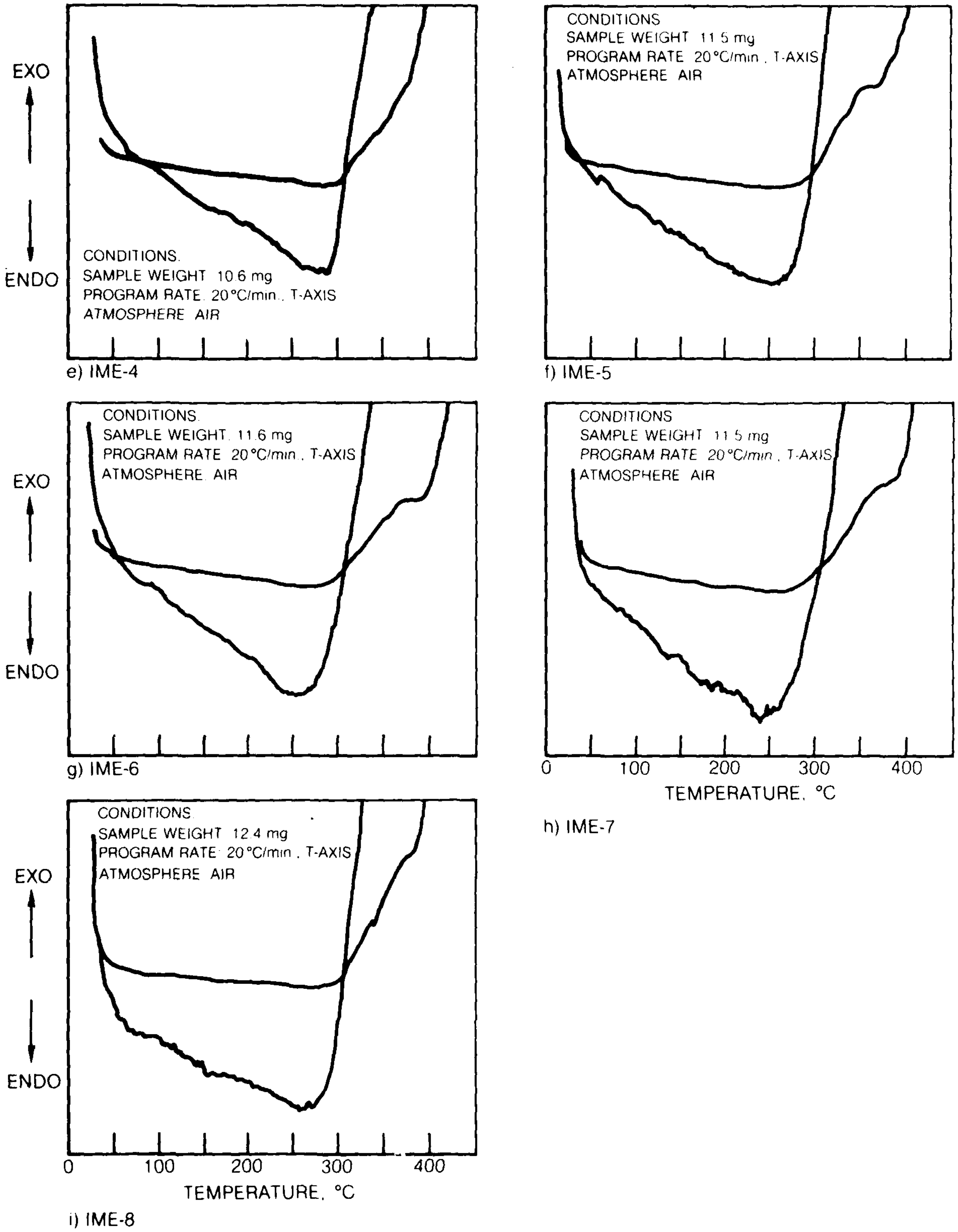
## DSC THERMOGRAMS OF CURED EPOXY RESINS




80-10-159-22

FIG. 17 (CONT'D)

## DSC THERMOGRAMS OF CURED EPOXY RESINS (CONTINUED)




80-10-159-23

## TMA OF IMIDE EPOXY RESINS

a)

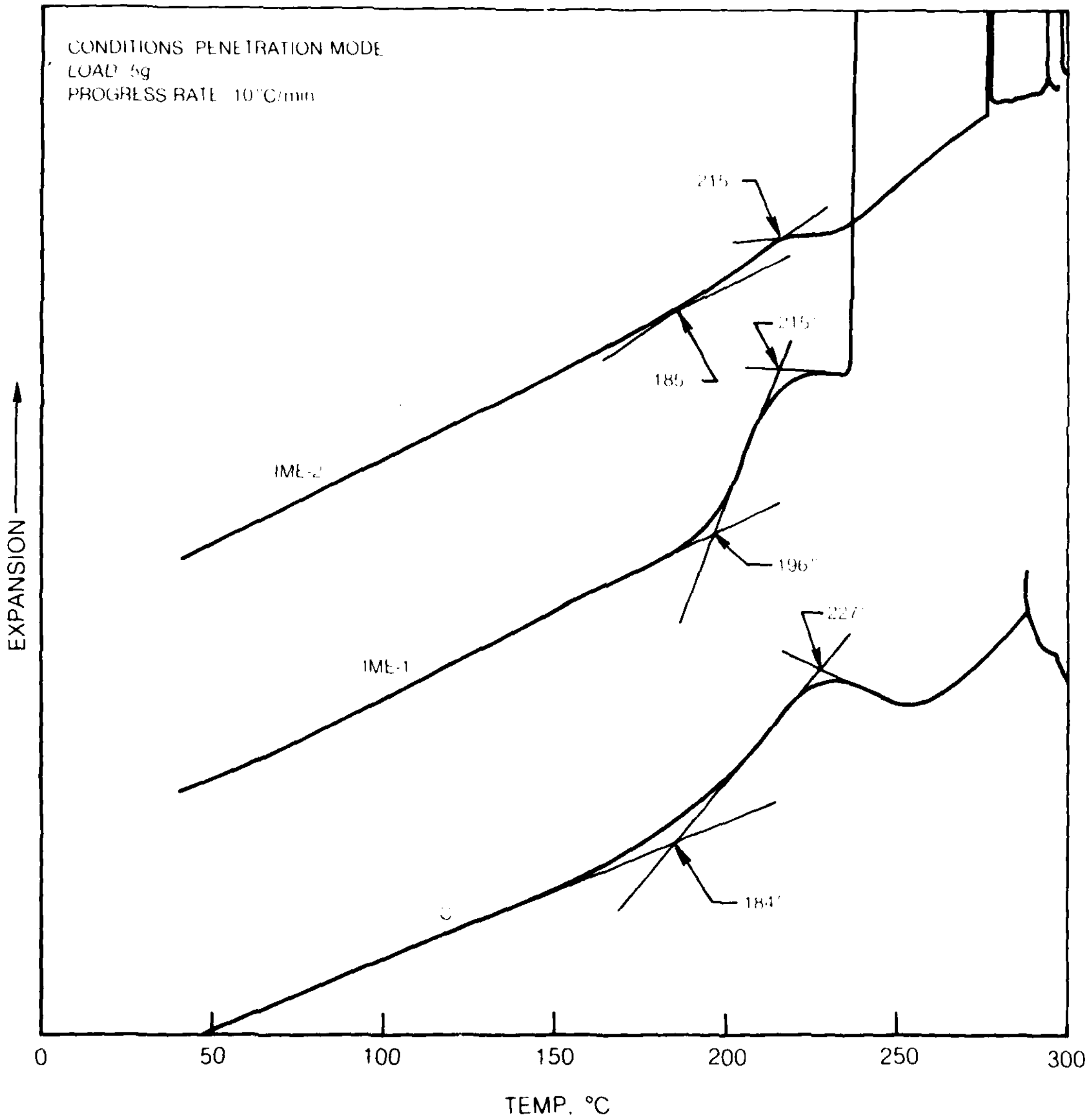
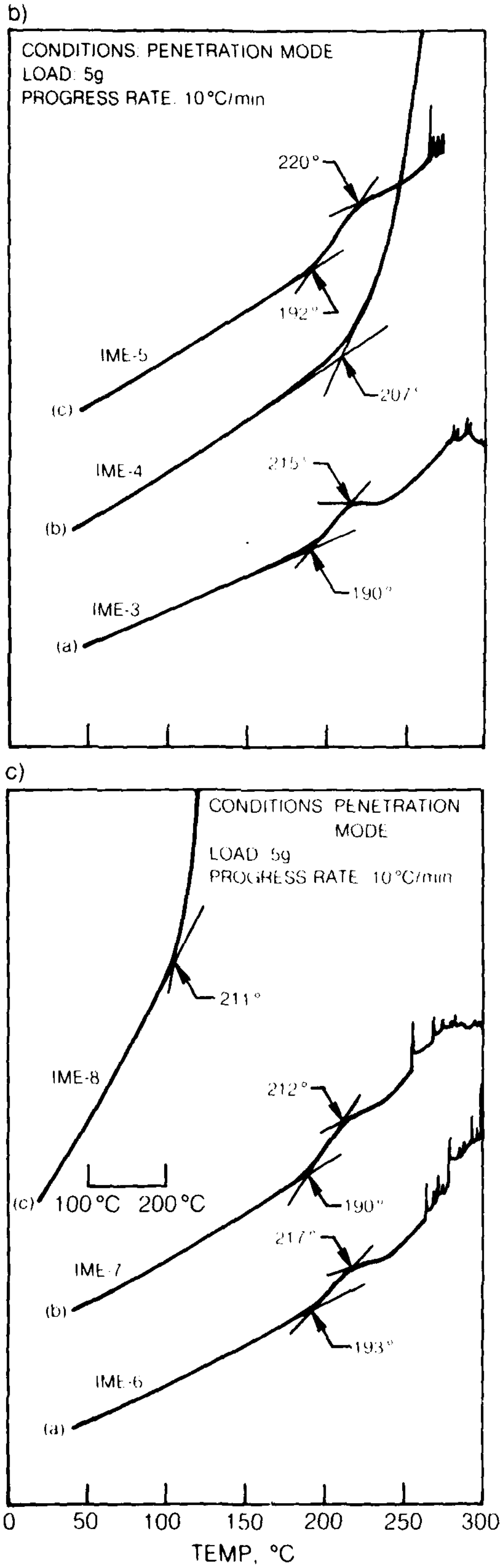


FIG. 18

## TMA OF IMIDE EPOXY RESINS




81-4 129-1

# THERMOGRAVIMETRIC ANALYSIS (TGA) OF CURED IME EPOXY RESINS

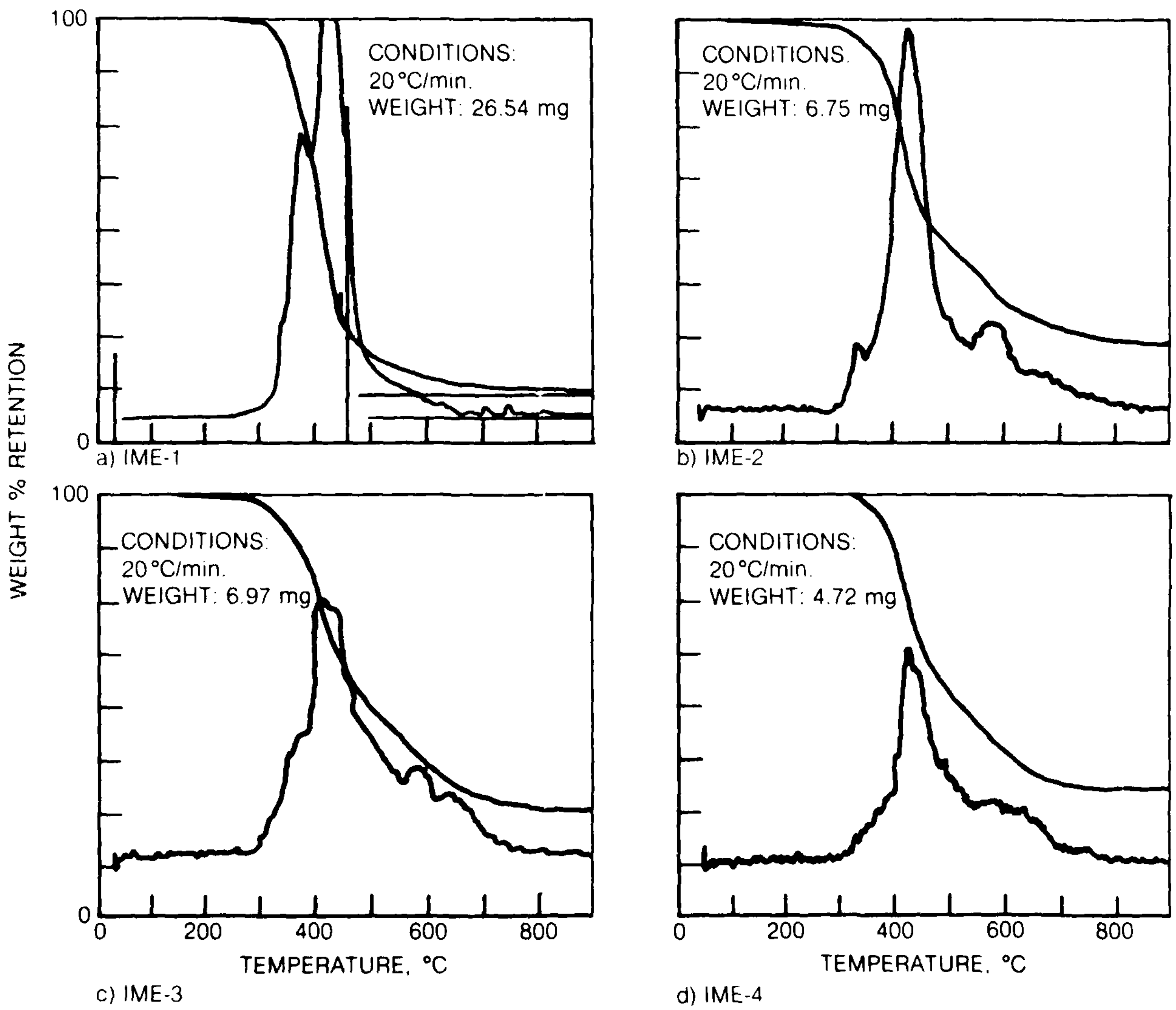
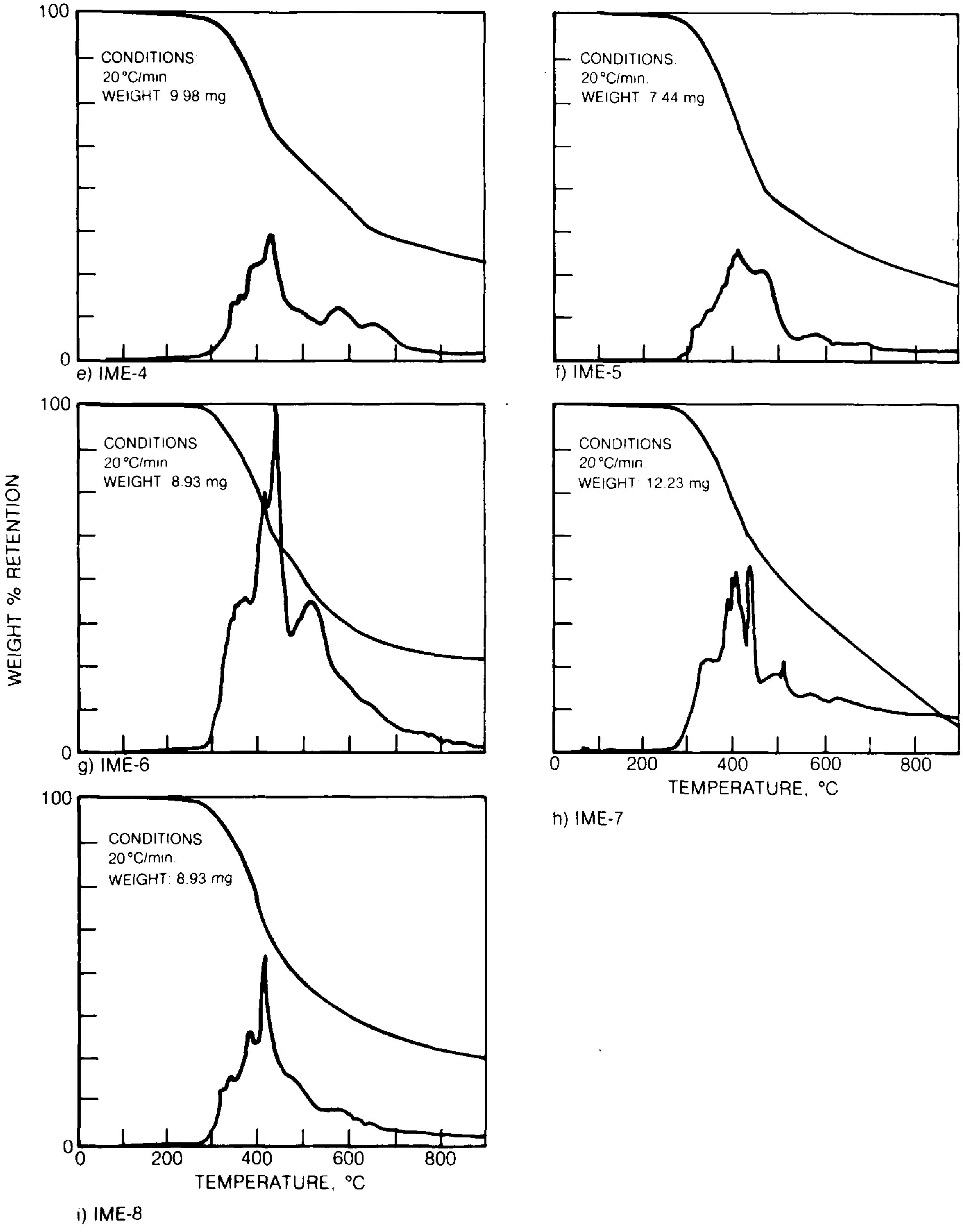


FIG. 19 (CONT.)

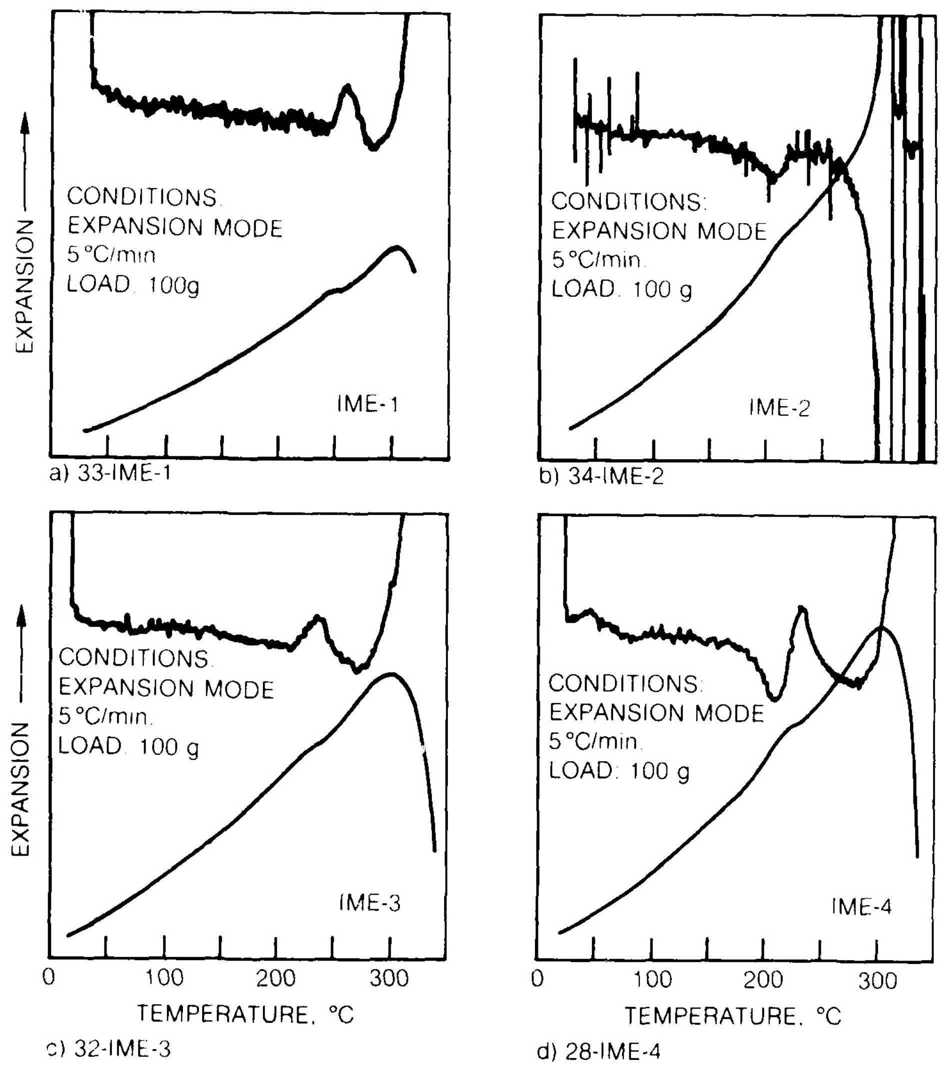
## THERMOGRAVIMETRIC ANALYSIS (TGA) OF CURED EPOXY RESINS (CONTINUED)




80–10–159–14

FIG. 20

## THERMOCHEMICAL ANALYSIS (TMA) OF CELION 6000/IME EPOXY RESIN COMPOSITES



AD-A102 122 UNITED TECHNOLOGIES RESEARCH CENTER EAST HARTFORD CT F/G 11/4
IMIDE MODIFIED EPOXY MATRIX RESIN.(U)
FEB 81 D A SCOLA, R H PATER NAS3-22032
UNCLASSIFIED UTRC/R81-914770-12 NASA-CR-165229 NL

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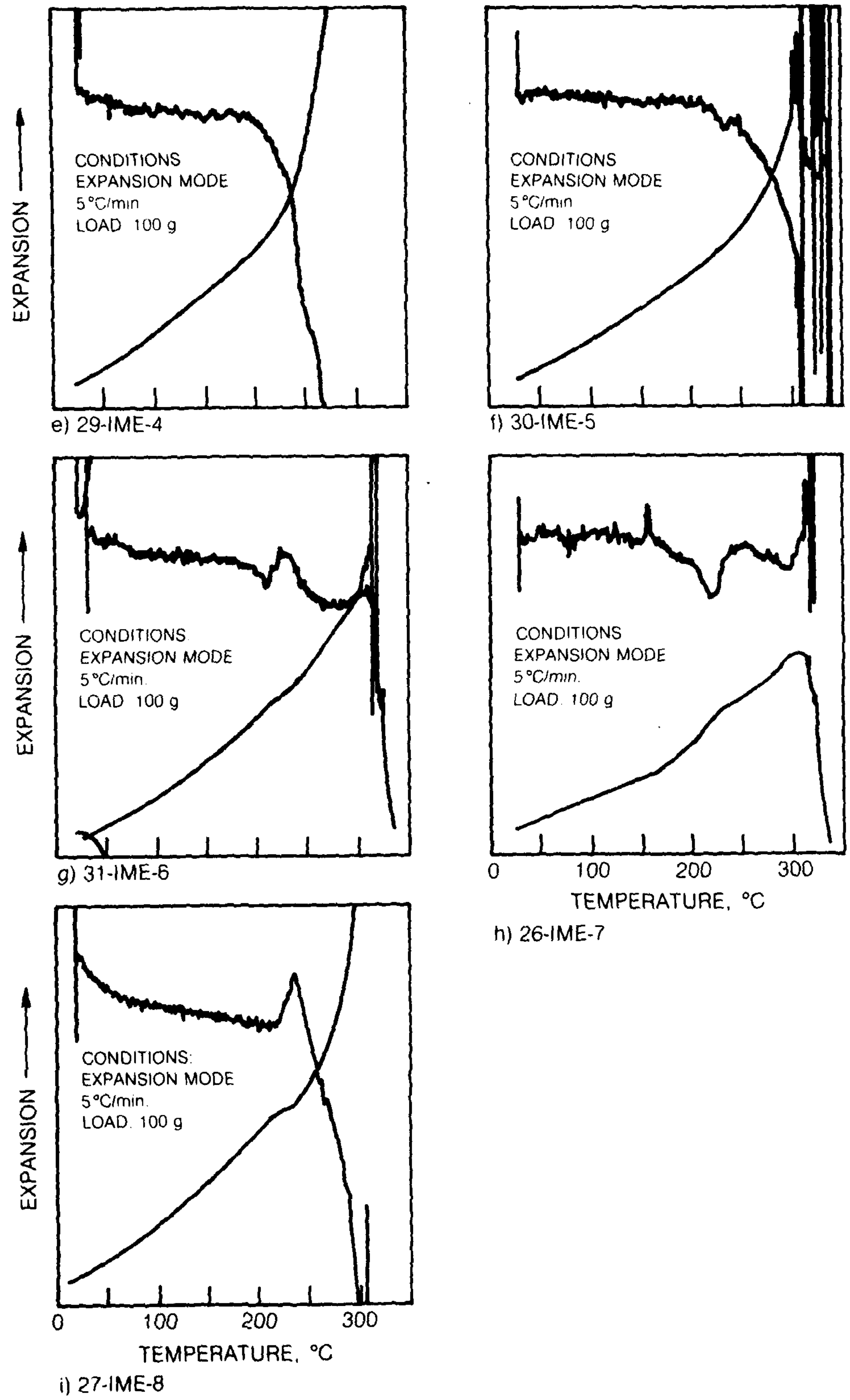
FIG. 20 (CONT'D)

**THERMOMECHANICAL ANALYSIS (TMA) OF CELION 6000 EPOXY RESIN COMPOSITES (CONTINUED)**




80-10-159-10

**THERMOGRAVIMETRIC ANALYSIS (TGA) OF CELION 6000/IME EPOXY RESIN COMPOSITES**

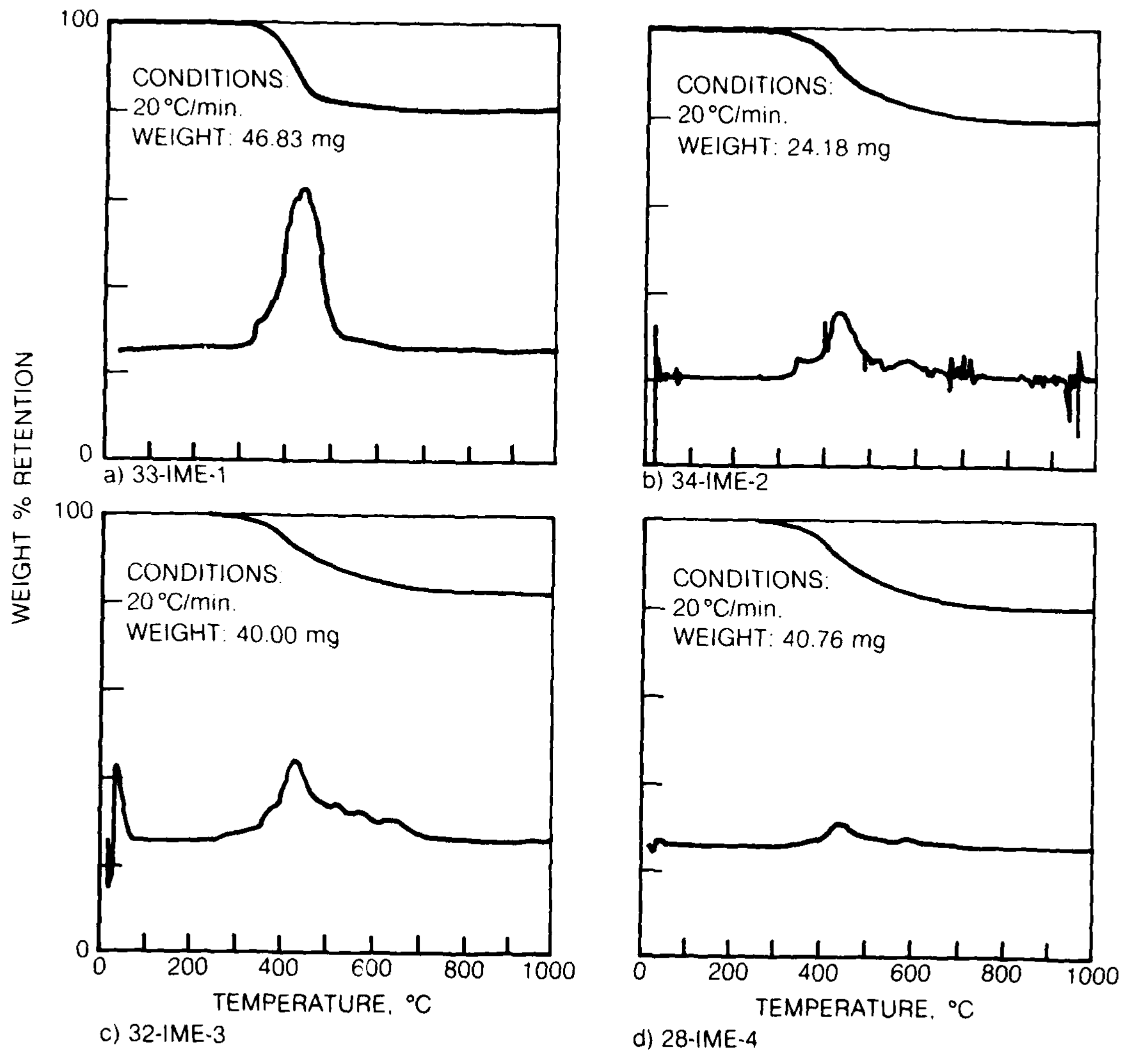
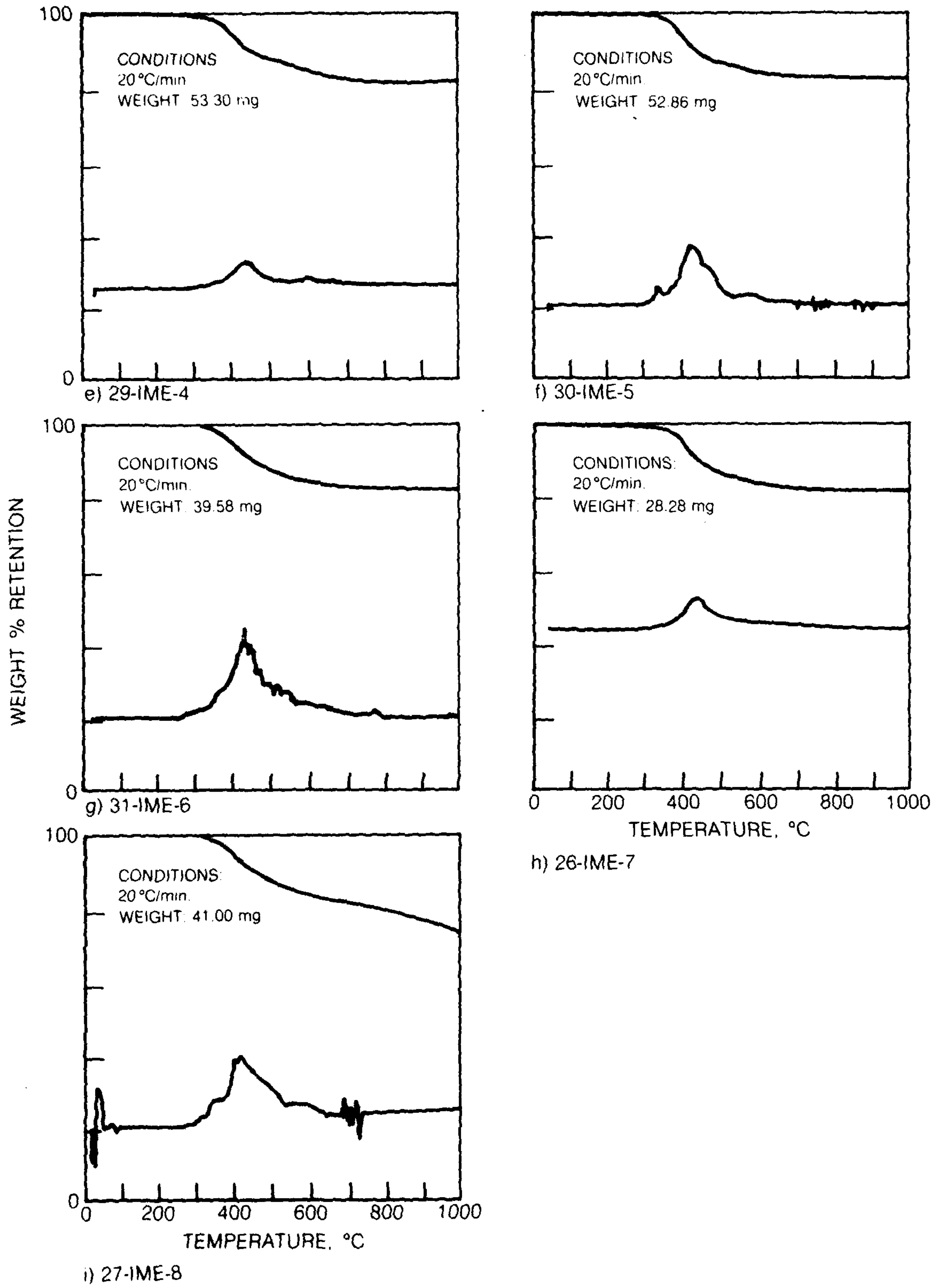


FIG. 21 (CONT'D)

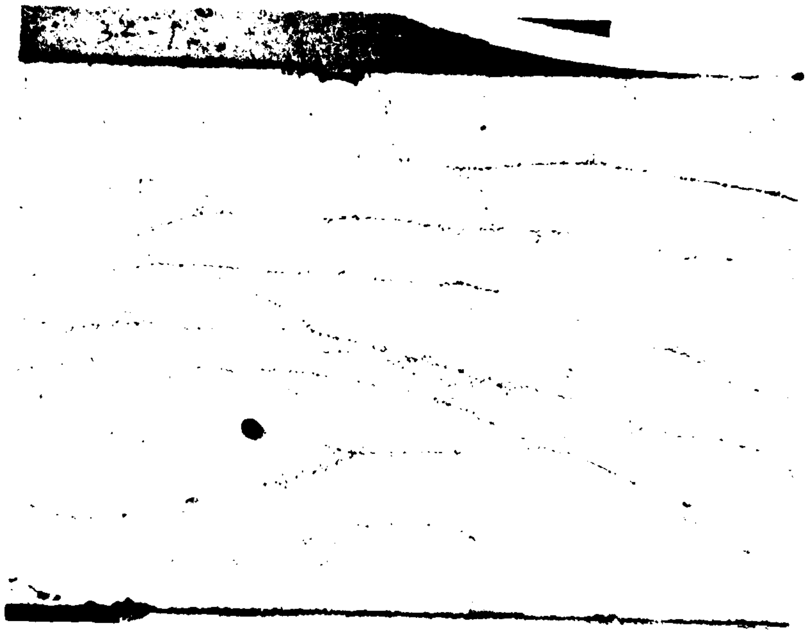
**THERMOGRAVIMETRIC ANALYSIS (TGA) OF CELION 6000 EPOXY RESIN COMPOSITES (CONTINUED)**




80-10-159-8

FIG. 22

## OPTICAL MICROGRAPHS OF CELION 6000/IME EPOXY RESIN COMPOSITES



a) 33-C

700μ

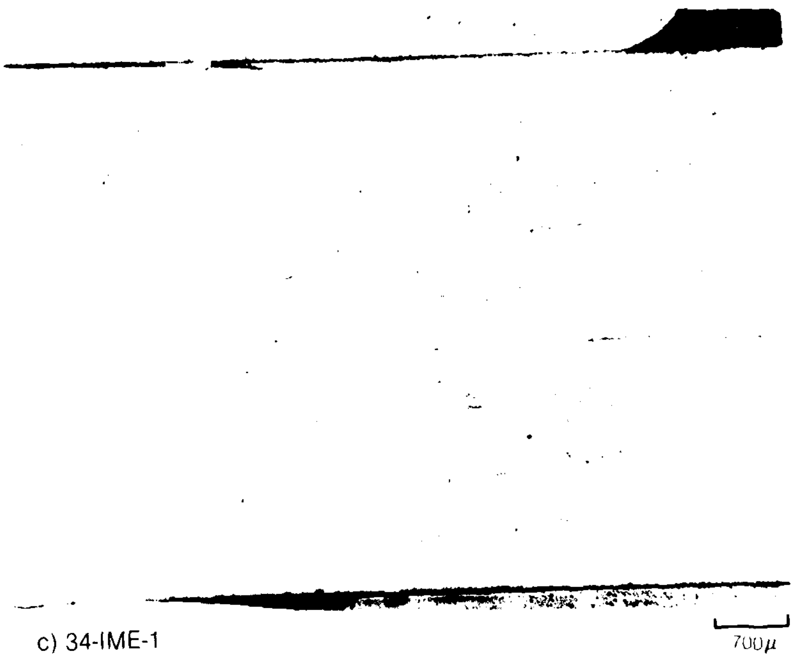
b) 20-IME-1

700μ

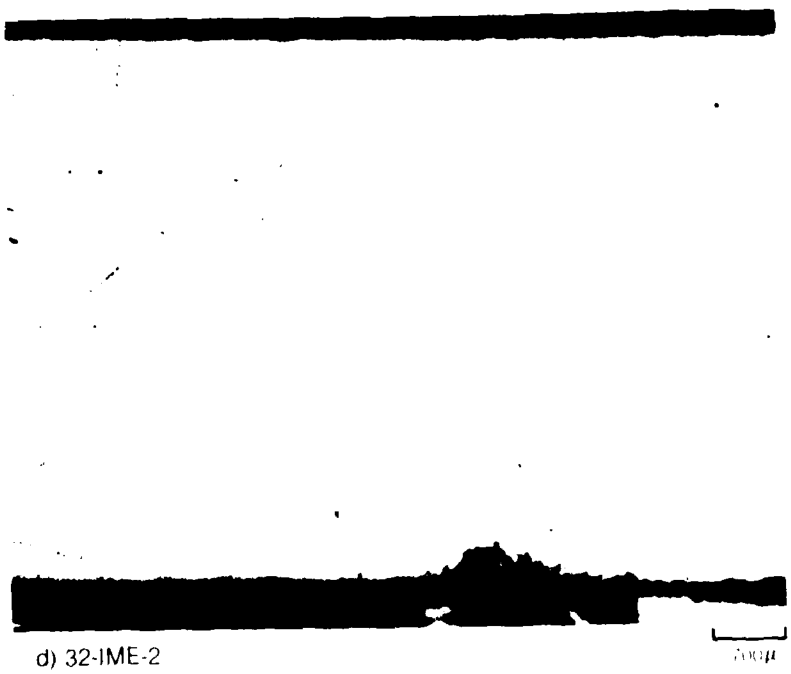
80-10-159-1

FIG. 22

# OPTICAL MICROGRAPHS OF CELION 6000 EPOXY RESIN COMPOSITES (CONTINUED)



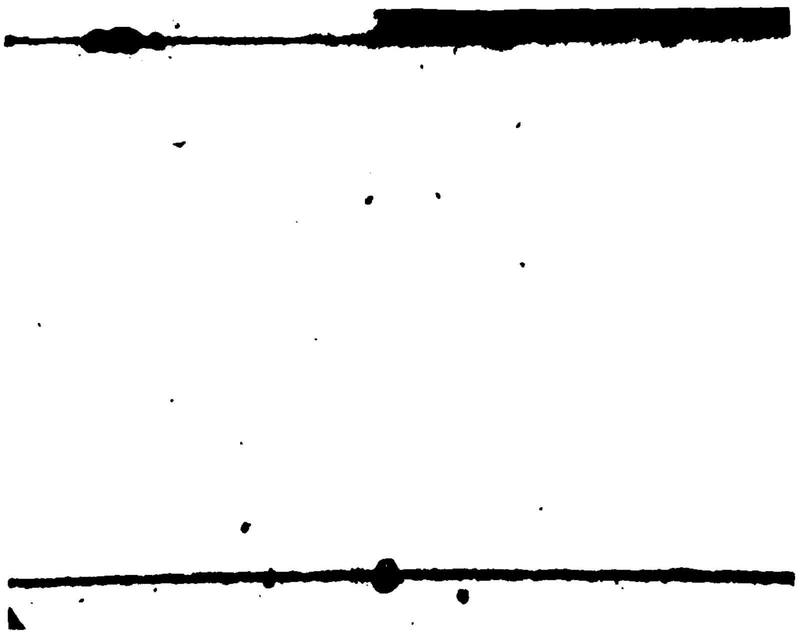

c) 34-IME-1




d) 32-IME-2

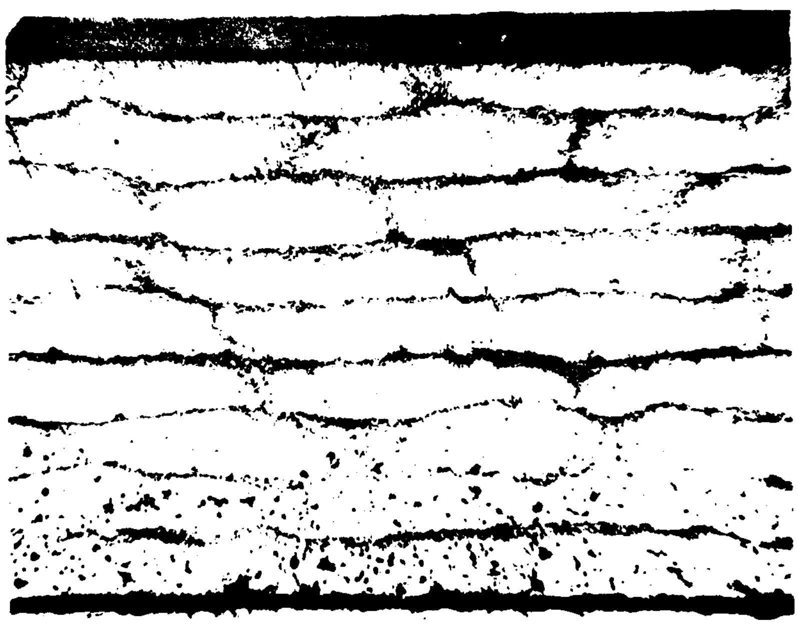
FIG. 22

# OPTICAL MICROGRAPHS OF CELION 6000 EPOXY RESIN COMPOSITES (CONTINUED)



e) 28-IME-3

700μ

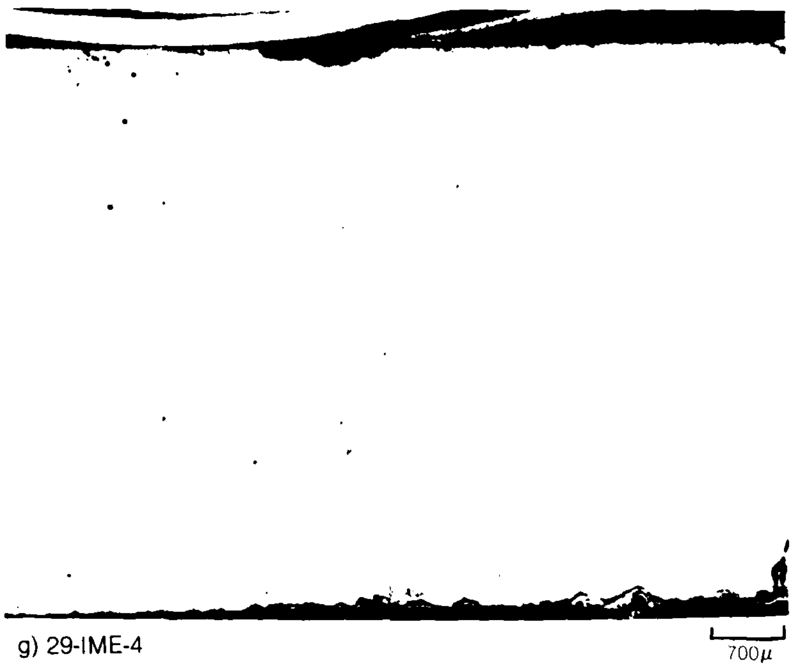


f) 23-IME-4

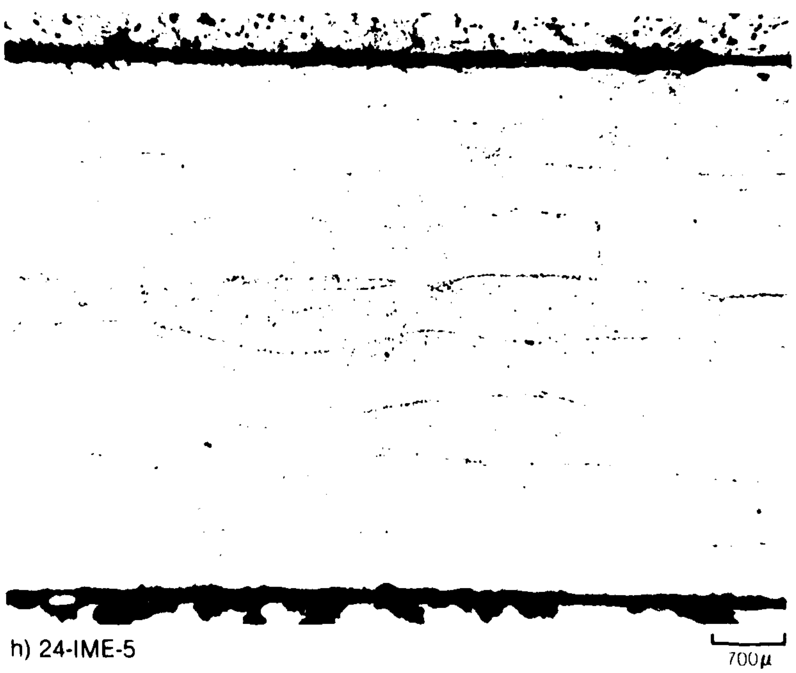
700μ

80-10-159-3

**OPTICAL MICROGRAPHS OF CELION 6000
EPOXY RESIN COMPOSITES (CONTINUED)**



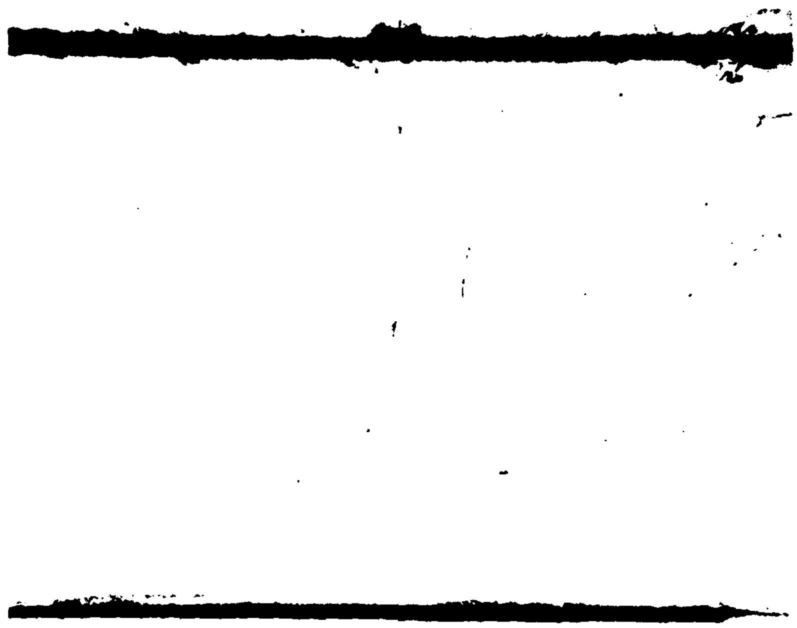

g) 29-IME-4



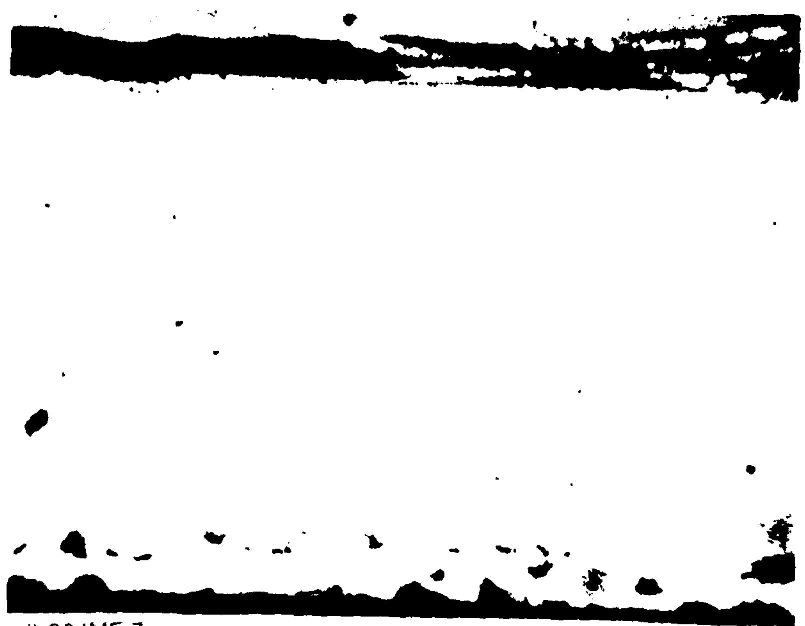

h) 24-IME-5

FIG. 22

**OPTICAL MICROGRAPHS OF CELION 6000 EPOXY RESIN COMPOSITE (CONTINUED)**



i) 31-IME-6 700μ

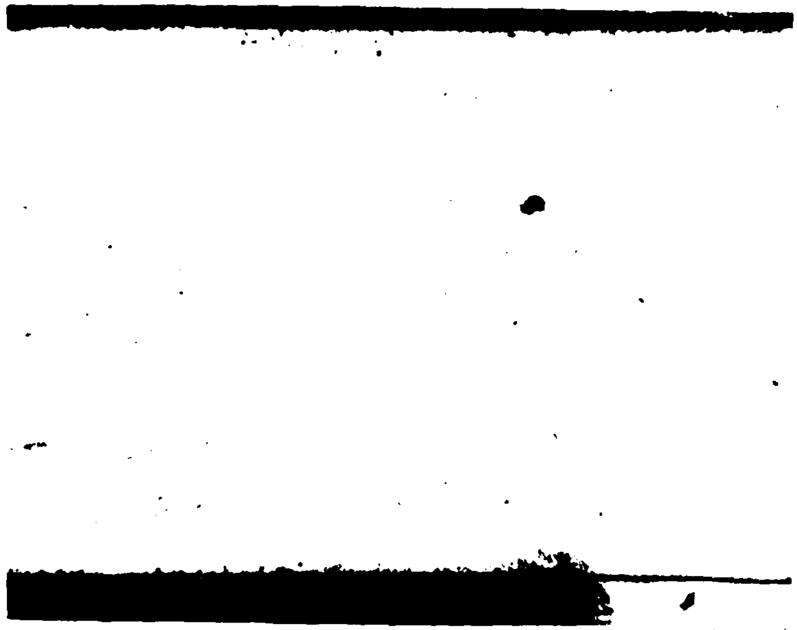


j) 26-IME-7 700μ

80-10-159-5

FIG. 22

**OPTICAL MICROGRAPHS OF CELION 6000/IME EPOXY RESIN COMPOSITES (CONTINUED)**



k) 30-IME-5

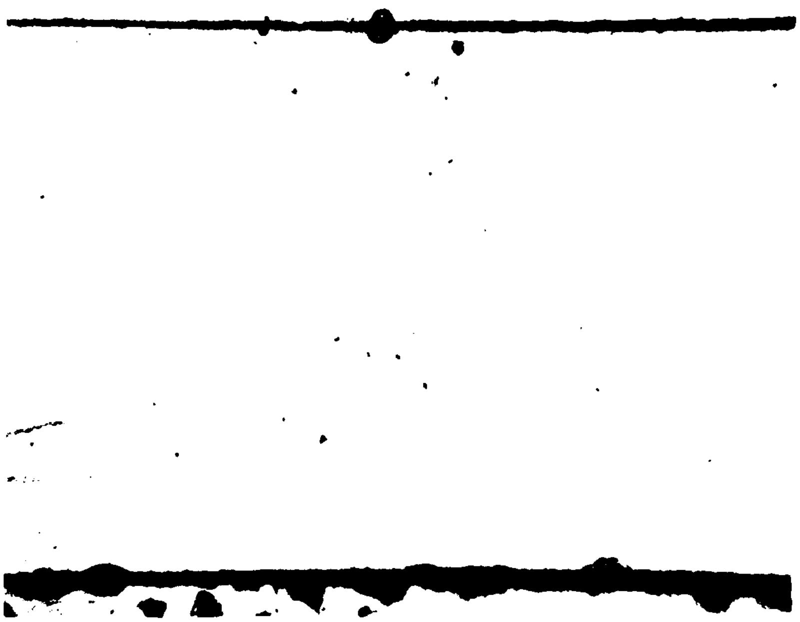
700μ

l) 25-IME-6

700μ

80–10–159–19

**OPTICAL MICROGRAPHS OF CELION 6000/IME EPOXY RESIN COMPOSITE (CONCLUDED)**



m) 27-IME-8

700μ

## EXPERIMENTAL SET-UP FOR AMPLITUDE MODULATED ULTRASONIC C-SCANS

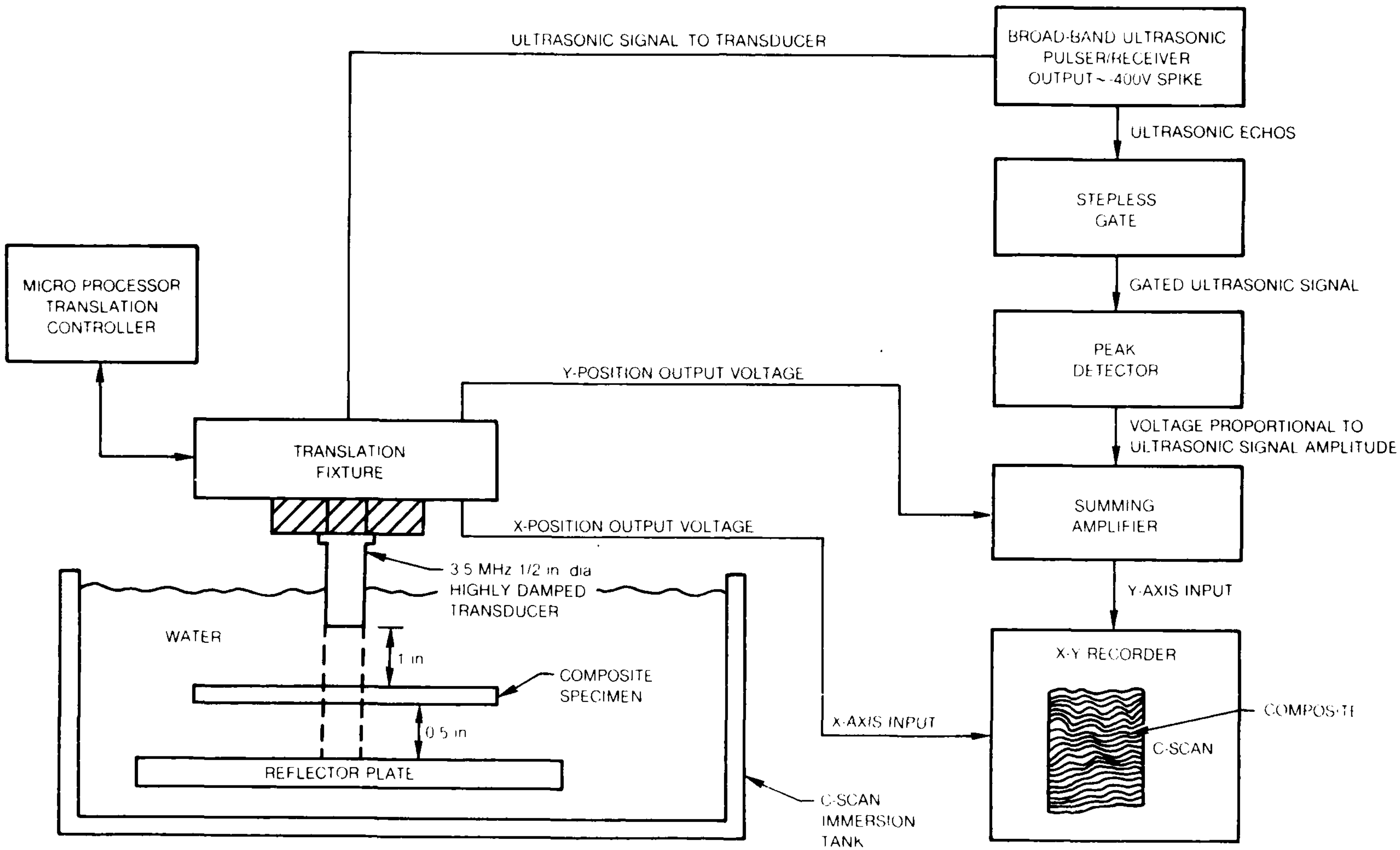



FIG 23

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